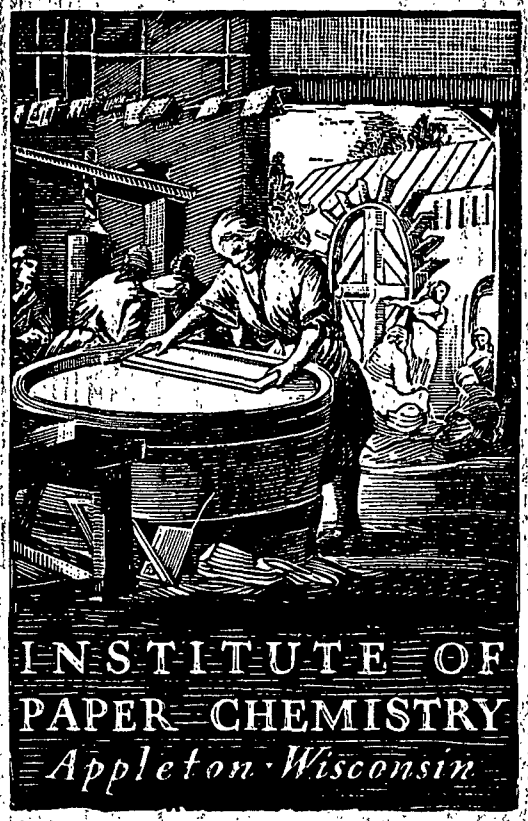

IMPROVED BONDING STRENGTH OF GROUNDWOOD FURNISHES

Project 2948

Report Three

A Progress Report

to

MEMBERS OF GROUP PROJECT 2948

April 10, 1974

THE INSTITUTE OF PAPER CHEMISTRY

Appleton, Wisconsin

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Appleton Papers, Inc. — Locks Mill

Blandin Paper Company

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International Paper Company

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THE INSTITUTE OF PAPER CHEMISTRY

Appleton, Wisconsin

IMPROVED BONDING STRENGTH OF GROUNDWOOD FURNISHES

SUMMARY

Part One of the extended program on Project 2948 was concerned with refinement of the jet/alkali process for aspen stone groundwood. The first step in the extended program involved modification of the jet cooker to accommodate groundwood consistencies up to 5%. A fresh supply of aspen stone groundwood was then procured and preliminary experiments were conducted at 230°F to compare the effects of jet cooking at 2 and 5% consistency at equal effective alkali concentration, i.e., 0.2%. The jet processed pulp was blended with bleached softwood kraft in a ratio of 80/20 groundwood/kraft and handsheets were formed under standardized conditions at pH 5. Comparisons were made with sheets formed from 100% kraft and from 50/50 and 80/20 blends of untreated groundwood and kraft. The results confirmed the effectiveness of the jet/alkali process for aspen stone groundwood and indicated that the concentration of alkali in solution is not the controlling factor in strength development since rather substantial differences were obtained at the same effective alkali concentration.

An examination of process variables was then made in which the groundwood was jet cooked at 5% consistency at temperatures of 190, 230, and 280°F at alkali addition levels of 0, 4, 6, and 8% based on fiber weight. Dwell time was controlled at either 6 or 13 seconds. In general, strength properties (breaking length, T.E.A., tensile stiffness, and folding endurance) increased as the alkali-addition level increased up to 6-8%. However, brightness declined with increase in alkali addition and reached a level at alkali additions in excess of 6%, which was lower than that afforded by the 80/20 controls containing groundwood processed at 2% consistency with 10% alkali. The intermediate processing

temperature offered the best balance in strength and optical properties and, hence, jet treatment at 230°F with 6% alkali was selected as optimum or near optimum considering all properties involved. The breaking length and tensile stiffness values of the 80/20 groundwood/kraft blend attained under these conditions were 87 and 106%, respectively, of those afforded by the 100% kraft controls. Further, the 80/20 blend approached or equalled the 50/50 untreated groundwood/kraft controls in T.E.A., folding endurance, and scattering coefficient while stretch, air permeability, and brightness were lower than the kraft-containing controls. Increasing the dwell time in the jet cooker from 6 to 13 seconds provided little or no advantage. Chemical analysis of the jet-cooked pulps revealed that combined alkali reaches a plateau level of approximately 40 mg/g at alkali additions of 6-8% regardless of the processing temperature or cooking time. This is the same level found in previous work at 2% fiber consistency with 5, 10, and 15% alkali (1).

While the jet/alkali process produced marked increases in strength, brightness was adversely affected. In efforts to offset this loss in brightness, several agents, including sodium sulfite, hydrogen peroxide (with sodium silicate), and sodium borohydride were added to the jet cooking liquor in separate experiments. Three percent of sodium sulfite and 0.1-0.5% of borohydride afforded modest increases in strength and brightness in the 80/20 groundwood/kraft handsheets, but the brightness level attained was lower than that of the untreated groundwood/kraft controls. Brightness loss was prevented by addition of 1.2-1.3% of peroxide although strength was reduced somewhat under these conditions. Increasing the dwell time in the cooker again failed to provide significant improvements.

Medium density bleaching experiments (12.5% consistency) were carried out on untreated and jet/alkali-treated groundwood utilizing 1 and 2% of hydrogen

peroxide. In contrast to the decline in strength associated with addition of peroxide to the jet cooker, medium density bleaching produced modest increases in most strength properties and the brightness loss due to jet/alkali processing was offset with less than 1% of peroxide. Bleaching with 2% of peroxide increased brightness in the 80/20 groundwood/kraft blend 18 units to a level of 71.4% which was only two units lower than that provided by the bleached uncooked groundwood/kraft controls. At the same time, breaking length reached 95% of the 100% kraft controls and T.E.A., tensile stiffness, and folding endurance exceeded the values provided by the 50/50 controls.

As part of the experimental program, a limited number of handsheets were routinely formed from 100% groundwood, primarily for brightness measurements. Other physical tests subsequently determined on a selected number of these papers revealed that, in some cases, the breaking length and tensile stiffness equalled or exceeded those of the 100% kraft controls. Bleached groundwood paper approached or exceeded the all-kraft controls in density, breaking length, tensile stiffness, opacity, and scattering coefficient. Further, the bleached groundwood paper exceeded the 50/50 untreated groundwood kraft controls in breaking length and tensile stiffness at equal brightness. In effect, addition of kraft pulp to the jet/alkali-cooked groundwood reduced breaking length in several cases and had no effect in several others. As would be expected, the all-groundwood papers tended to provide reduced stretch and tear properties.

Examination of untreated and jet-treated aspen stone groundwood with a scanning electron microscope indicates that the mechanism of strength improvement in the jet/alkali process is probably one of increased bonded area resulting from the rupture of fibers along their length and the ability of these modified

fibers to open up and span a relatively large number of unreacted fiber elements. This was contrasted to refiner and thermomechanical treatment of wood chips in which case strength is reportedly improved through an increase in the long fiber fraction (2).

INTRODUCTION

This is Progress Report Three on Project 2948 concerned with means of improving the bonding strength of groundwood furnishes. More specifically, the present report describes results obtained in Part One of the extended program directed at refinement of the jet/alkali system for aspen stone groundwood.

Work previously reported on this project (1) demonstrated that improved strength properties can be attained in groundwood-containing furnishes by subjecting the groundwood component to the jet/alkali process. This process involves passing an alkaline suspension of the groundwood through a steam injection unit under controlled conditions of fiber consistency, time, temperature, and alkali concentration. The responsiveness of the system was found to depend upon the wood species utilized. Aspen groundwood was found to be very responsive as indicated by the fact that mixtures containing 80% of the treated groundwood and 20% softwood kraft approached or exceeded the tensile strength of the 100% kraft controls. A softwood groundwood was found to be somewhat less responsive. Brightness was reduced in the jet/alkali process but the reduction was offset in the hardwood furnish by incorporating 2.5% of peroxide into the treatment liquor. Yields ranged from 93 to 98% which is somewhat higher than those reported in the literature for alkaline treatments of longer duration (3). Presumably, the very limited exposure of the pulp to alkali in the jet treatment reduces the loss of the more soluble components.

While the effects of alkali concentration and temperature were explored to some extent, they were not optimized in the work reported. The fiber consistency was necessarily maintained at approximately 2% because of physical limitations of the jet cooker. However, this consistency is well below the normal

groundwood processing level and, hence, the concentration of alkali and other chemicals required in the jet/alkali process may not have been optimum. Therefore, emphasis in Part One of the extended program was concerned with determining the minimum amounts of reactants required to achieve the desired strength and optical properties at higher fiber consistency. The second part of the program will be directed at means of improving the jet/alkali system for softwood groundwood.

EXPERIMENTAL

GENERAL PROCEDURES

Pulp Supplies and Treatments

A fresh supply of aspen unbleached stone groundwood was procured from Kimberly-Clark Corporation. The pulp was dewatered to 25.7% consistency and stored at 40°F with preservative. For use in handsheets, the pulp was resuspended in tap water at either two or five percent consistency using a British disintegrator.

The bleached softwood kraft pulp (Rayonier WBS-W) used in the earlier work was again utilized in the present program. In preparation for handsheet making, the dry lap pulp was soaked overnight in tap water and then refined to 680 ml Schopper-Riegler freeness in a "5 pound" Valley beater. The Canadian freeness was 345 ml. The refined pulp was dewatered to 21.3% solids and then stored at 40°F with preservative. For use in handsheets, portions of the pulp were redispersed at 2.5% consistency in tap water by subjecting the diluted stock to 300 counts in a British disintegrator followed by further dilution to 2.0% consistency.

Modification of Jet Cooker

The jet cooker described in Progress Report One was modified to accommodate 5% consistency groundwood as required in the current program. A sketch of the modified unit is presented in Fig. 1. The major changes involved installation of a 1-hp variable speed drive, substitution of a 1-gal stainless steel hopper in place of the dual intake line, reduction of the inner diameter of the cooking chamber and installation of a throttling valve. Temperature and pressure gages were installed between the cooking chamber and

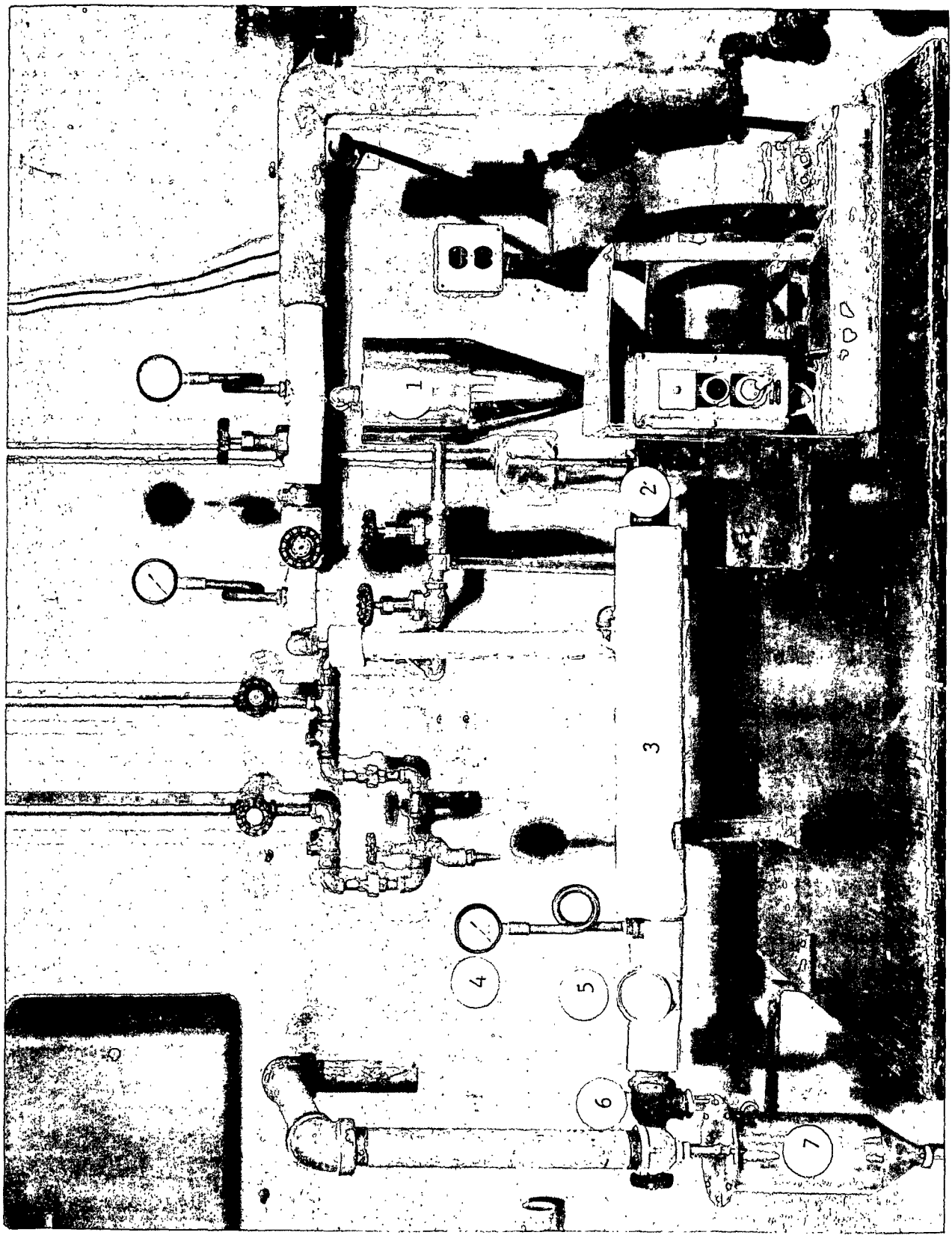

Figure 1. Jet Cooker. 1. Hopper; 2. Moyno Pump; 3. Jet Reaction Chamber; 4. Pressure Indicator; 5. Temperature Indicator; 6. Throttling Valve; 7. Separator

the throttling valve and a water supply line was added to feed the hopper. The diameter of the cooking chamber was reduced in order to prevent or minimize retention of the high consistency pulp at the inlet to the jet. As a result of these modifications, the effective volume was changed such that the dwell time at the same pumping rate was reduced to four seconds compared to six seconds in the earlier version. The dwell time was increased to a maximum of 13 seconds by changing pulleys in the drive system.

Operation of Jet Cooker

The general procedure for operating the jet cooker was as follows:

Room temperature water was fed to the hopper and the steam pressure adjusted to provide the desired temperature. When steady state conditions were attained and a low level of water remained in the hopper either six liters of pulp at 2% consistency or four liters of 5% stock treated with the desired amount of reactants was added. Approximately 3.0-3.5 liters of treated pulp was collected after discarding the first several hundred ml. The final flow through the jet was also discarded. The collected pulp was cooled to room temperature within approximately 10 minutes and a sample withdrawn for determination of residual NaOH and other reactants. The major portion of the collected material was then neutralized with dilute hydrochloric acid to pH 5. The pulp suspension was sampled at this point for consistency, Canadian Standard freeness, and yield.

Handsheet Preparations

The equivalent of 32 g (o.d. basis) of the neutralized groundwood from the jet cooker was blended with 8 g (o.d. basis) of the bleached kraft pulp to provide an 80/20 blend by weight for handsheets. The blended pulp was diluted to 0.5% consistency with tap water and the pH was adjusted to 5 with dilute sulfuric acid. Handsheets equivalent to 2.5 g (o.d. basis) were

then formed in the 8 x 8-inch Noble & Wood mold in the same manner described in Progress Report Two with the exception that 12 sheets were prepared per set instead of eight. Two sheets from each batch of jet-cooked groundwood were prepared for brightness measurements.

Testing and Analysis

Handsheets were preconditioned at 20% RH, 70°F and then at 50% RH and 73°F prior to testing according to TAPPI procedures. Tests included basis weight, thickness, density, breaking length, stretch, tensile energy absorption (T.E.A.), extensional stiffness, tear factor (single sheet tear), folding endurance, air permeability, opacity, and scattering coefficient. Brightness was measured on unequilibrated paper within 24 hours of preparation.

The treated pulp suspensions were tested for residual NaOH, yield, and for residual peroxide, where appropriate, according to the procedures given in the appendix.

SPECIFIC STUDIES

Examination of Process Variables

The first series of experiments which formed a base line for subsequent studies in the extended program was primarily directed at the effects of fiber consistency in the jet/alkali process at a cooking temperature of 230°F. In this direction, comparisons were made of the groundwood jet cooked at 2% consistency with 10% of NaOH (based on fiber) and at 5% consistency with 4% of NaOH. The effective concentration of alkali in water in both cases was 0.2%. The effect of dwell time was also examined to the extent that groundwood was cooked at the new dwell time of the modified jet cooker (4 seconds) and at the original dwell time of six seconds. The processed pulp was blended 80/20 with the bleached

kraft pulp and handsheets formed in the manner previously described. Reference controls were also prepared from 100% kraft and from 80/20 and 50/50 blends of untreated groundwood and kraft. Results are presented in Tables I and II.

A second series of tests which was directed at establishing the minimum amount of alkali required to achieve the optimum balance in strength and optical properties was then carried out at 5% consistency. This study examined the effects of incremental increases in alkali concentration and changes in cooking temperature and dwell time. The alkali concentration was varied from 0 to 8% (based on fiber) at each of three jet processing temperatures, i.e., 190, 230, and 280°F. The dwell time was maintained at 6 seconds in each of these separate series. The series producing the best balance in strength and optical properties was subsequently repeated at a 13-second dwell time. Physical test results are recorded in Table III. Yield and residual alkali data are presented in Table IV. Sheet properties as a function of alkali concentration are shown graphically in Fig. 2-23. Free and combined alkali as functions of alkali addition level are presented in Fig. 24.

Examination of Brightness Loss Inhibitors

In order to offset a notable decline in brightness associated with the jet/alkali process, a number of bleaching agents or brightness loss inhibitors were incorporated into the pulp prior to treatment in the jet. This work was limited to the 230° treatment with 6% alkali or, in effect, to the conditions selected on the basis of the results obtained in the previous series. Sodium sulfite was added in amounts of 1, 3, and 7% (based on fiber). Hydrogen peroxide was used at 1, 2, and 3% (based on fiber) in combination with 5% sodium silicate and at 1 and 3% in combination with 10% of sodium silicate. Sodium borohydride was utilized at 0.1 and 0.5% (based on fiber) on an as is

TABLE I

BASE-LINE STUDIES WITH FRESH SAMPLE OF ASPEN STONE GROUNDWOOD

Set No.	Description	Fiber Consistency in Jet, %	NaOH Added, % based on fiber	Dwell Time in Jet Cooker, sec	Basis Weight, g/M²	S.E.	Thickness, µm	S.E.	Density, g/cc	Breaking Length, km	S.E.	T.E.A., g cm/sq cm	S.E.	Stretch, %	S.E.	Tensile Stiffness, kg/cm	S.E.
1	Control, 100% kraft	--	--	--	61.4	0.42	114.3	1.14	0.537	5.10	0.10	89.17	3.40	4.01	0.09	341	4.3
2	Control, 50/50 untreated groundwood/kraft	--	--	--	61.9	0.05	145.3	0.95	0.426	3.28	0.06	42.11	2.21	2.80	0.10	241	4.4
3	Control, 80/20 untreated groundwood/kraft	--	--	--	63.0	0.09	160.5	0.52	0.393	2.61	0.01	24.21	0.67	1.99	0.04	216	3.3
4	80/20 Treated groundwood/kraft	2	10	4	62.8	0.13	127.5	0.51	0.493	4.19	0.06	39.23	2.72	2.06	0.11	335	5.2
5	80/20 Treated groundwood/kraft	5	4	4	62.8	0.20	142.7	0.52	0.440	3.41	0.07	35.21	1.13	2.23	0.03	274	9.0
6	80/20 Treated groundwood/kraft	2	10	6	62.4	0.28	123.4	0.63	0.506	4.58	0.02	39.80	0.82	1.92	0.04	377	4.6
7	80/20 Treated groundwood/kraft	5	4	6	62.4	0.24	137.7	0.52	0.453	3.64	0.01	29.91	1.73	1.83	0.10	301	9.8
8	Reference No. 87[a] (80/20 treated groundwood/kraft)	2	10	6	64.2		--		0.51	4.91		42.2		1.9		405	

Set No.	Description	Fiber Consistency in Jet, %	NaOH Added, % based on fiber	Dwell Time in Jet Cooker, sec	Tear Factor, (single sheet)	S.E.	MIT Folding Endurance, double folds	S.E.	Bendtsen Air Permeability, ml/min	S.E.	Groundwood Brightness, %	S.E.	Paper Brightness, %	S.E.	Opacity, %	S.E.	Scattering Coeff., sq cm/g	S.E.
1	Control, 100% kraft	--	--	--	124.1	5.4	367	30.3	255	15.5	--		86.4	0.15	71.5	0.39	304	4.9
2	Control, 50/50 untreated groundwood/kraft	--	--	--	78.2	2.3	20	1.5	399	12.4	--		69.4	0.12	86.6	0.20	522	5.2
3	Control, 80/20 untreated groundwood/kraft	--	--	--	48.2	1.2	4	0.2	431	11.9	64.5	0.12	66.5	0.13	91.4	0.10	649	2.2
4	80/20 Treated groundwood/kraft	2	10	4	53.1	1.6	18	0.9	78	2.8	53.3	0.16	56.2	0.10	90.1	0.18	521	4.4
5	80/20 Treated groundwood/kraft	5	4	4	56.1	1.5	8	0.4	208	3.6	55.9	0.37	59.5	0.16	92.1	0.13	592	3.8
6	80/20 Treated groundwood/kraft	2	10	6	56.4	3.1	18	1.0	72	1.9	52.8	0.11	55.6	0.11	90.1	0.16	513	4.0
7	80/20 Treated groundwood/kraft	5	4	6	52.0	1.2	10	0.5	151	6.1	56.7	0.17	58.6	0.14	91.7	0.07	570	2.1
8	Reference No. 87[a] (80/20 treated groundwood/kraft)	2	10	6	--		--		47		63.1		52.4		91.3		--	

[a]From Progress Report Two — this set contained first sample of aspen stone groundwood.

TABLE II

GROUNDWOOD SUSPENSION DATA FOR BASE-LINE STUDIES

Cook No.	Fiber Consistency, %	NaOH Added, % based on fiber	NaOH Concn. in Soln., %	Processing Temp., °F	Dwell Time, sec	pH of Pulp Slurry		Total Solids of Pulp Slurry, %	Sol. Solids of Pulp Slurry, %	Yield, %	Free NaOH		Combined NaOH		Total NaOH	
						Before Cook	After Cook				Meq/ 100 ml	Mg/g	Meq/ 100 ml	Mg/g	Mg/g	% Accounted for
1	2.0	10.0	0.2	230	4	12.7	12.5	2.10	0.33	96.6	2.18	41.5	2.02	38.5	80.0	88.0
2	5.0	4.0	0.2	230	4	12.2	11.1	4.74	0.38	97.4	0.22	1.85	3.73	31.5	33.4	87.0
3	2.0	10.0	0.2	230	6	12.6	12.5	2.03	0.38	93.1	2.40	47.2	2.05	40.4	87.6	96.2
4	5.0	4.0	0.2	230	6	11.9	11.1	4.73	0.41	96.7	0.25	2.11	3.90	33.0	35.1	91.4

TABLE III

EXAMINATION OF PROCESS VARIABLES IN THE JET-ALKALI PROCESS

Set No.	Description	Fiber Consistency in Jet, %	NaOH Added, % based on fiber	Dwell Time in Jet Cooker, sec	Processing Temp., °F	Basis Weight, g/M²	S.E.	Thickness, µm	S.E.	Density, g/cc	Breaking Length, km	S.E.	T.E.A., g cm/sq cm	S.E.	Stretch, %	S.E.	Tensile Stiffness, kg/cm	S.E.
1	Controls, 100% kraft	--	--	--	--	61.4	0.42	114.3	1.14	0.537	5.10	0.10	89.17	3.40	4.01	0.09	341	4.3
2	Controls, 50/50 untreated groundwood/kraft	--	--	--	--	61.9	0.05	145.3	0.95	0.426	3.28	0.06	42.11	2.21	2.80	0.10	241	4.4
3	Controls, 80/20 untreated groundwood/kraft	--	--	--	--	63.0	0.09	160.5	0.52	0.393	2.61	0.01	24.21	0.67	1.99	0.04	216	3.3
6	Controls, 80/20 treated groundwood/kraft	2	10	6	230	62.4	0.28	123.4	0.63	0.506	4.58	0.02	39.80	0.82	1.92	0.04	377	4.6
9	80/20 Groundwood/kraft	5	0	6	230	62.6	0.14	157.5	0.81	0.398	2.72	0.05	20.21	0.88	1.67	0.04	233	2.9
10	" " "	5	4	6	230	62.4	0.24	137.7	0.52	0.453	3.64	0.01	29.91	1.73	1.83	0.10	301	9.8
11	" " "	5	6	6	230	63.5	0.12	122.4	0.95	0.519	4.42	0.09	38.31	1.91	1.89	0.07	360	6.8
12	" " "	5	8	6	230	61.5	0.13	114.3	0.81	0.538	4.66	0.08	33.68	1.98	1.67	0.06	365	4.5
13	" " "	5	0	6	280	63.2	0.12	159.5	0.51	0.396	2.56	0.07	23.27	1.41	1.93	0.10	214	7.0
14	" " "	5	4	6	280	61.3	0.21	133.6	1.02	0.459	3.28	0.07	24.72	1.87	1.70	0.09	272	4.5
15	" " "	5	6	6	280	65.1	0.16	125.0	0.95	0.521	4.04	0.07	36.96	2.89	1.91	0.10	341	5.1
16	" " "	5	8	6	280	61.5	0.13	117.3	0.51	0.524	4.16	0.05	36.25	2.24	1.92	0.08	323	2.7
17	" " "	5	0	6	190	63.0	0.41	163.1	1.25	0.386	2.80	0.04	20.57	1.37	1.71	0.08	227	1.5
18	" " "	5	4	6	190	63.5	0.10	141.2	1.02	0.450	3.63	0.03	31.19	1.22	1.95	0.05	278	2.9
19	" " "	5	6	6	190	62.1	0.15	126.5	0.51	0.491	4.25	0.20	35.07	1.16	1.83	0.03	324	7.0
20	" " "	5	8	6	190	62.6	0.17	119.9	0.95	0.522	4.62	0.08	37.79	2.23	1.88	0.09	346	6.9
21	" " "	5	0	13	230	64.2	0.18	157.5	0.81	0.408	2.69	0.04	23.92	1.10	1.92	0.06	228	3.7
22	" " "	5	4	13	230	64.1	0.28	133.1	0.62	0.482	3.61	0.06	29.61	1.59	1.80	0.08	311	7.3
23	" " "	5	6	13	230	63.3	0.10	121.4	0.51	0.521	4.40	0.04	36.35	1.18	1.86	0.05	361	5.1
24	" " "	5	8	13	230	64.2	0.19	118.9	0.51	0.540	4.12	0.02	30.03	0.51	1.64	0.03	361	5.6

TABLE III (Continued)

EXAMINATION OF PROCESS VARIABLES IN THE JET-ALKALI PROCESS

Set No.	Description	Fiber Consistency in Jet, %	NaOH Added, % based on fiber	Dwell Time in Jet Cooker, sec	Processing Temp., °F	Tear Factor, (single sheet)	S.E.	MIT Folding Endurance, double folds	S.E.	Bendtsen Air Permeability, ml/min	S.E.	Groundwood Brightness, %	S.E.	Paper Brightness, %	S.E.	Opacity, %	S.E.	Scattering Coeff., sq cm/g	S.E.
1	Controls, 100% kraft	--	--	--	--	124.1	5.4	367	30.3	255	15.5	—		86.4	0.15	71.5	0.39	304	4.9
2	Controls, 50/50 untreated groundwood/kraft	--	--	--	--	78.2	2.3	20	1.5	399	12.4	--		69.4	0.12	86.6	0.20	522	5.2
3	Controls, 80/20 untreated groundwood/kraft	--	--	--	--	48.2	1.2	4	0.2	431	11.9	64.5	0.12	66.5	0.13	91.4	0.10	649	2.2
6	Controls, 80/20 treated groundwood/kraft	2	10	6	230	56.4	3.1	18	1.0	72	1.9	52.8	0.11	55.6	0.11	90.1	0.16	513	4.0
9	80/20 Groundwood/kraft	5	0	6	230	51.1	1.8	5	0.2	571	12.7	63.0	0.11	64.4	0.09	92.2	0.16	651	5.8
10	" " "	5	4	6	230	52.0	1.2	10	0.5	151	6.1	56.7	0.17	58.6	0.14	91.7	0.07	570	2.1
11	" " "	5	6	6	230	53.2	1.3	19	1.4	88	1.4	52.9	0.09	55.7	0.07	90.2	0.13	511	3.3
12	" " "	5	8	6	230	53.0	1.1	30	2.2	64	1.3	50.2	0.12	53.2	0.09	90.0	0.23	486	5.4
13	" " "	5	0	6	280	55.4	3.5	4	0.2	472	6.3	63.6	0.12	65.1	0.11	92.2	0.14	658	4.3
14	" " "	5	4	6	280	56.7	1.7	9	0.8	191	3.7	57.9	0.17	59.3	0.08	92.2	0.17	602	5.7
15	" " "	5	6	6	280	54.1	1.1	19	1.1	75	2.0	54.4	0.10	56.8	0.06	90.0	0.33	523	8.1
16	" " "	5	8	6	280	56.5	1.3	15	0.9	68	1.4	50.2	0.17	53.2	0.08	90.5	0.16	493	4.3
17	" " "	5	0	6	190	51.4	3.1	4	0.2	512	11.1	62.3	0.34	64.2	0.15	92.8	0.18	672	7.2
18	" " "	5	4	6	190	55.4	3.0	11	0.5	160	5.5	55.9	0.19	58.7	0.07	92.6	0.27	585	7.5
19	" " "	5	6	6	190	52.2	2.6	25	1.4	87	2.2	53.0	0.08	55.6	0.13	90.7	0.21	513	5.9
20	" " "	5	8	6	190	55.9	1.6	34	2.7	66	1.1	49.9	0.11	52.2	0.15	91.6	0.13	500	3.7
21	" " "	5	0	13	230	48.0	0.6	4	0.2	474	13.2	60.3	0.13	62.1	0.17	93.4	0.26	649	9.3
22	" " "	5	4	13	230	54.0	1.8	12	0.7	171	3.0	54.0	0.16	56.7	0.18	92.8	0.22	566	7.5
23	" " "	5	6	13	230	51.8	1.3	21	1.0	72	1.6	50.1	0.24	53.2	0.35	92.1	0.10	515	5.0
24	" " "	5	8	13	230	50.8	0.9	27	1.6	64	2.2	47.9	0.09	51.2	0.10	91.6	0.22	492	6.5

TABLE IV

GROUNDWOOD SUSPENSION DATA — EXAMINATION OF PROCESS VARIABLES
(5% Fiber consistency)

Cook No.	NaOH Added, % based on fiber	NaOH Concn. in Soln., %	Proc. Temp., °F	Dwell Time, sec	pH of Pulp Slurry		Total Solids of Pulp Slurry, %	Sol. Solids of Pulp Slurry, %	Yield, %	Free NaOH		Combined NaOH		Total NaOH	
					Before Cook	After Cook				Meq/ 100 ml	Mg/g	Meq/ 100 ml	Mg/g	Mg/g	% Accounted for
5	None	--	230	6	7.4	7.4	4.64	0.04	98.9	--	--	0.25	2.1	2.1	--
4	4.0	0.2	230	6	11.9	11.1	4.73	0.41	96.7	0.25	2.11	3.90	33.0	35.1	91.4
6	6.0	0.3	230	6	12.5	12.2	4.76	0.51	97.1	1.37	11.5	4.83	40.6	52.1	92.0
7	8.0	0.4	230	6	12.5	12.5	4.96	0.72	95.5	3.32	26.8	4.93	39.7	66.7	90.0
8	None	--	280	6	7.4	7.8	4.50	0.05	98.8	--	--	0.25	2.2	2.2	--
9	4.0	0.2	280	6	12.2	11.5	4.49	0.35	97.6	0.31	2.76	3.70	32.9	35.7	93.0
10	6.0	0.3	280	6	12.5	12.3	4.45	0.45	97.8	1.50	13.5	4.20	37.7	51.2	90.4
11	8.0	0.4	280	6	12.6	12.5	4.59	0.58	97.6	3.12	27.2	4.53	39.5	66.7	90.0
12	None	--	190	6	7.3	7.3	4.53	0.06	98.7	--	--	0.22	1.94	1.94	--
13	4.0	0.2	190	6	12.3	11.6	4.59	0.32	98.4	0.41	3.57	3.64	31.7	35.3	91.9
14	6.0	0.3	190	6	12.6	12.3	4.65	0.55	95.9	1.61	13.8	4.39	37.8	51.6	91.2
15	8.0	0.4	190	6	12.7	12.6	4.77	0.67	96.1	3.66	30.8	5.00	41.9	72.7	98.1
16	None	--	230	13	7.3	7.0	4.43	0.06	98.6	--	--	0.18	1.63	1.63	--
17	4.0	0.2	230	13	12.1	11.4	4.62	0.36	97.6	0.23	1.99	3.87	33.5	35.4	92.2
18	6.0	0.3	230	13	12.5	12.3	4.66	0.55	95.9	1.37	11.8	4.53	38.9	50.8	89.7
19	8.0	0.4	230	13	12.7	12.6	4.75	0.65	96.4	3.37	26.3	4.93	41.5	67.8	91.5

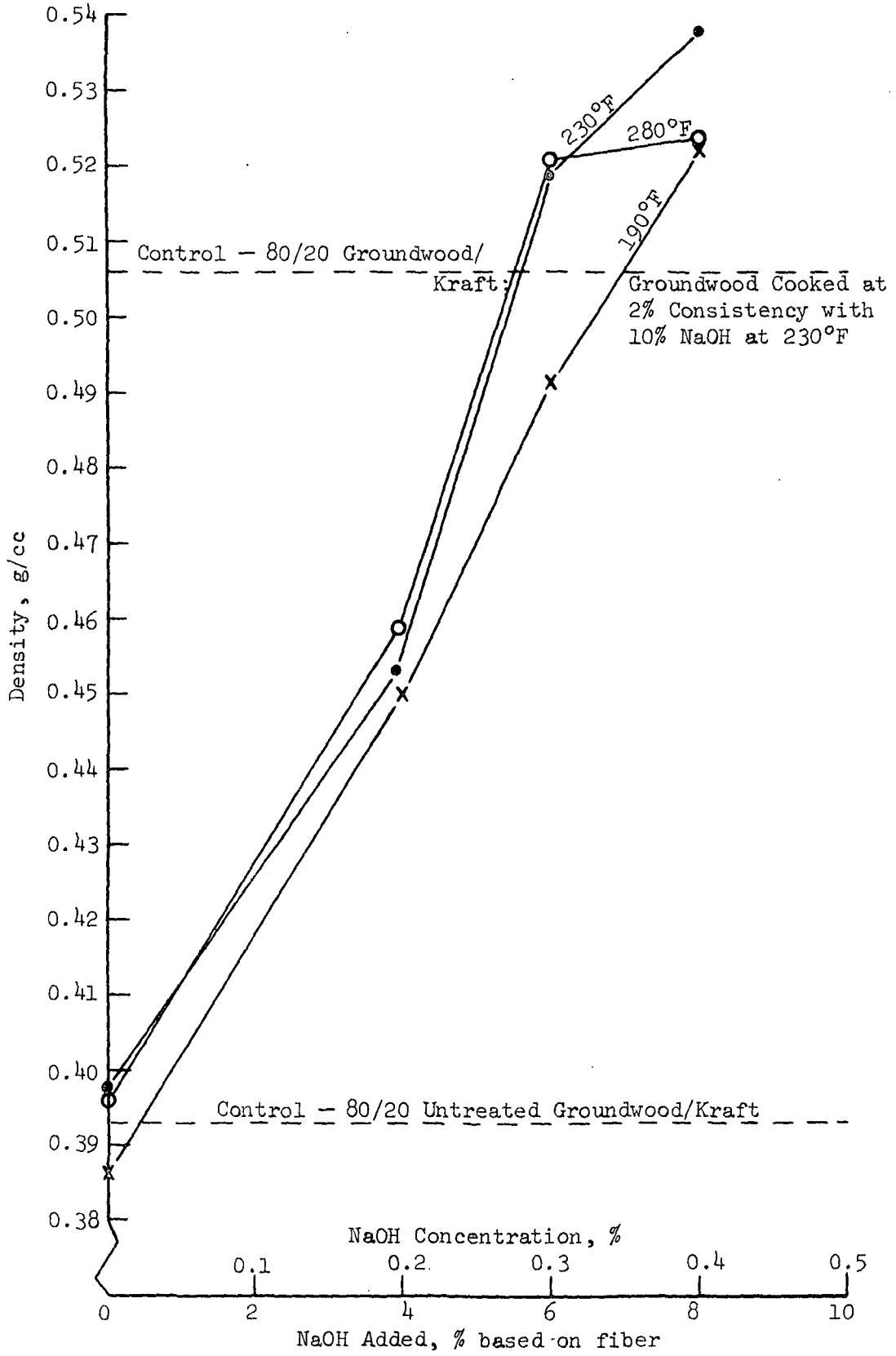

Figure 2. The Effect of Alkali Level and Temperature on Sheet Density (80/20 Blends of Groundwood/Kraft; Groundwood Cooked at 5% Consistency; 6-Sec Dwell Time)

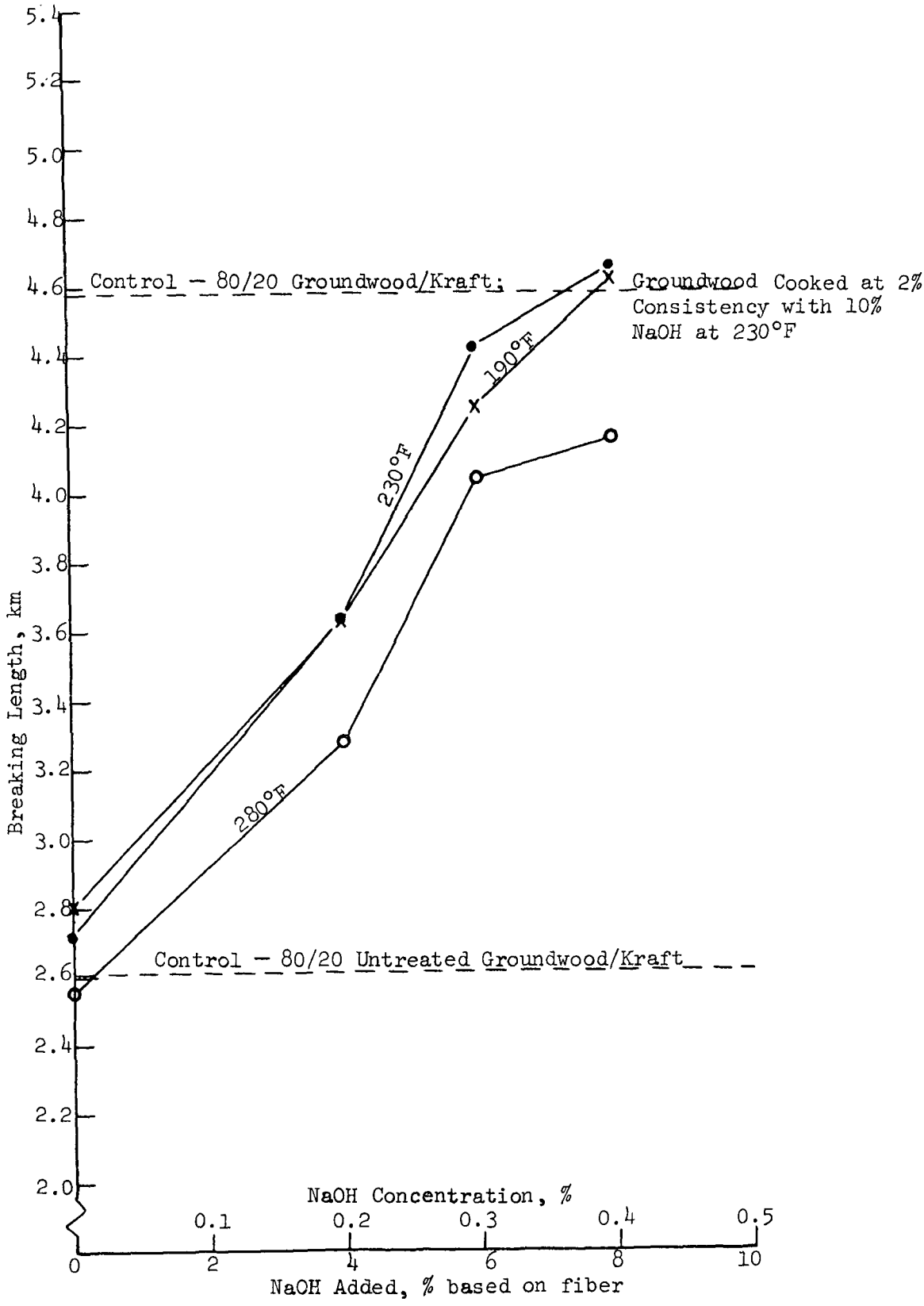

Figure 3. The Effect of Alkali Level and Temperature on Breaking Length (80/20 Blends of Groundwood/Kraft; Groundwood Cooked at 5% Consistenty; 6-Sec Dwell Time)

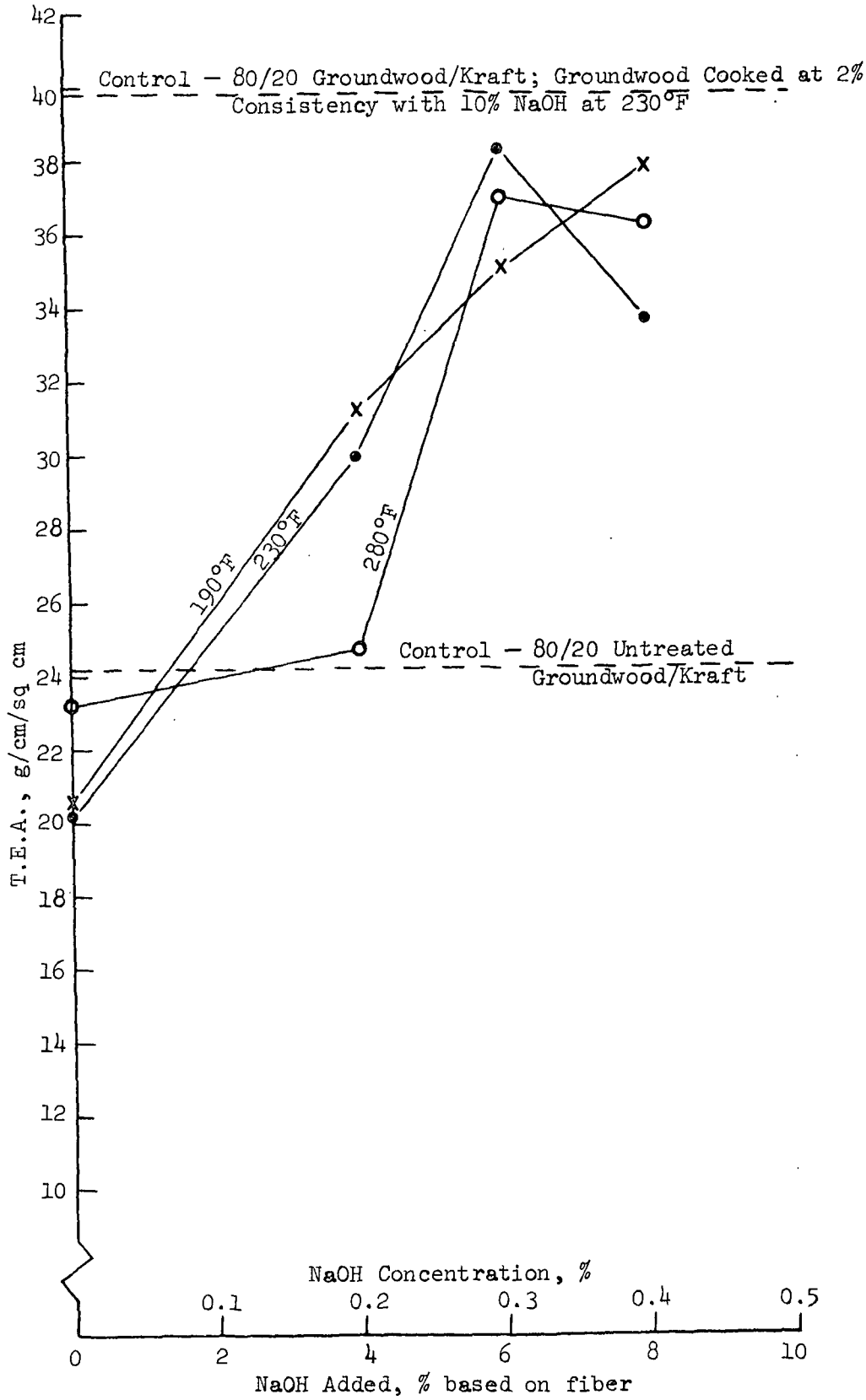

Figure 4. The Effect of Alkali Level and Temperature on Tensile Energy Absorption (80/20 Blends of Groundwood/Kraft; Groundwood Cooked at 5% Consistency; 6-Sec Dwell Time)

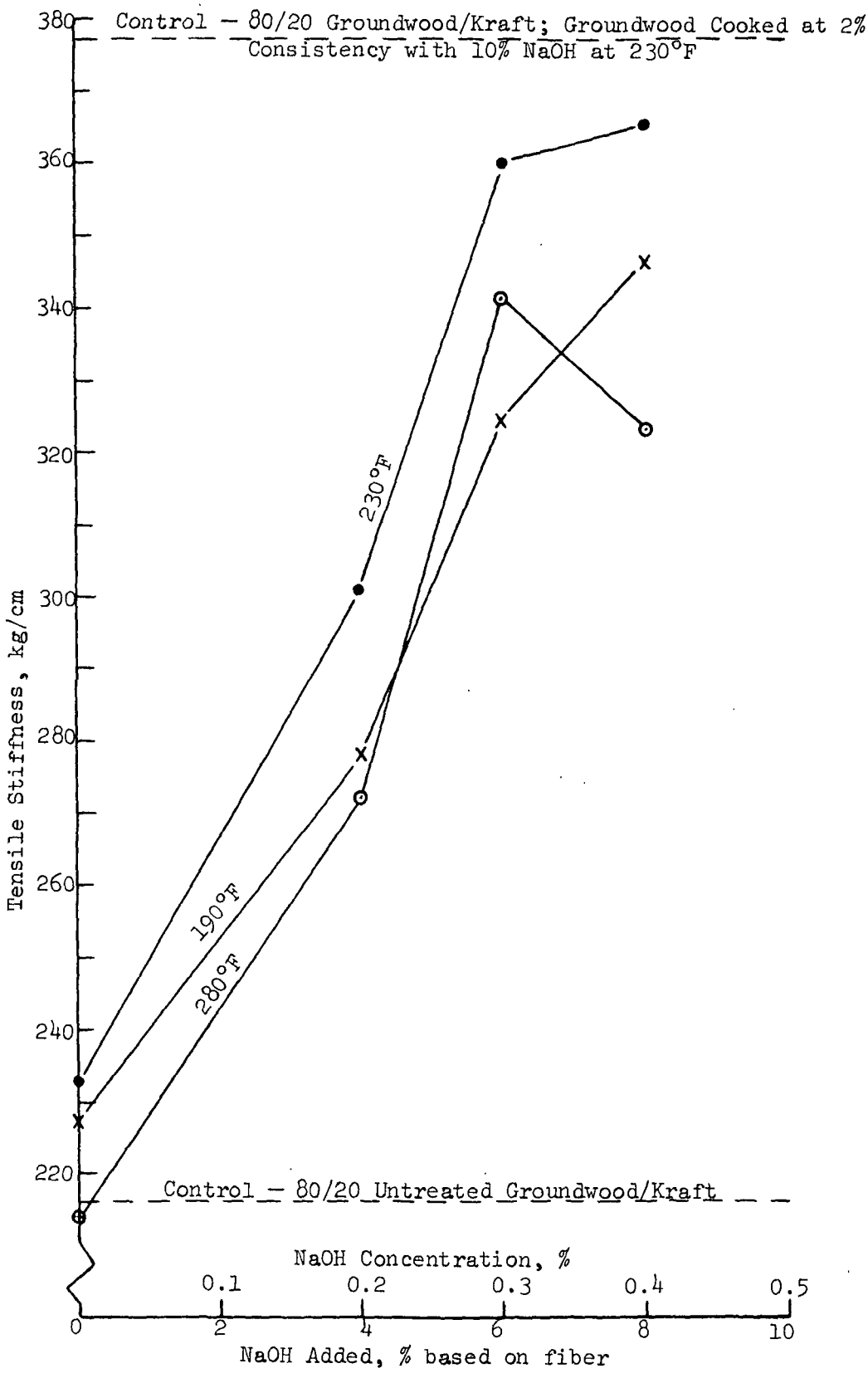

Figure 5. The Effect of Alkali Level and Temperature on Tensile Stiffness (80/20 Blends of Groundwood/Kraft; Groundwood Cooked at 5% Consistency; 6-Sec Dwell Time)

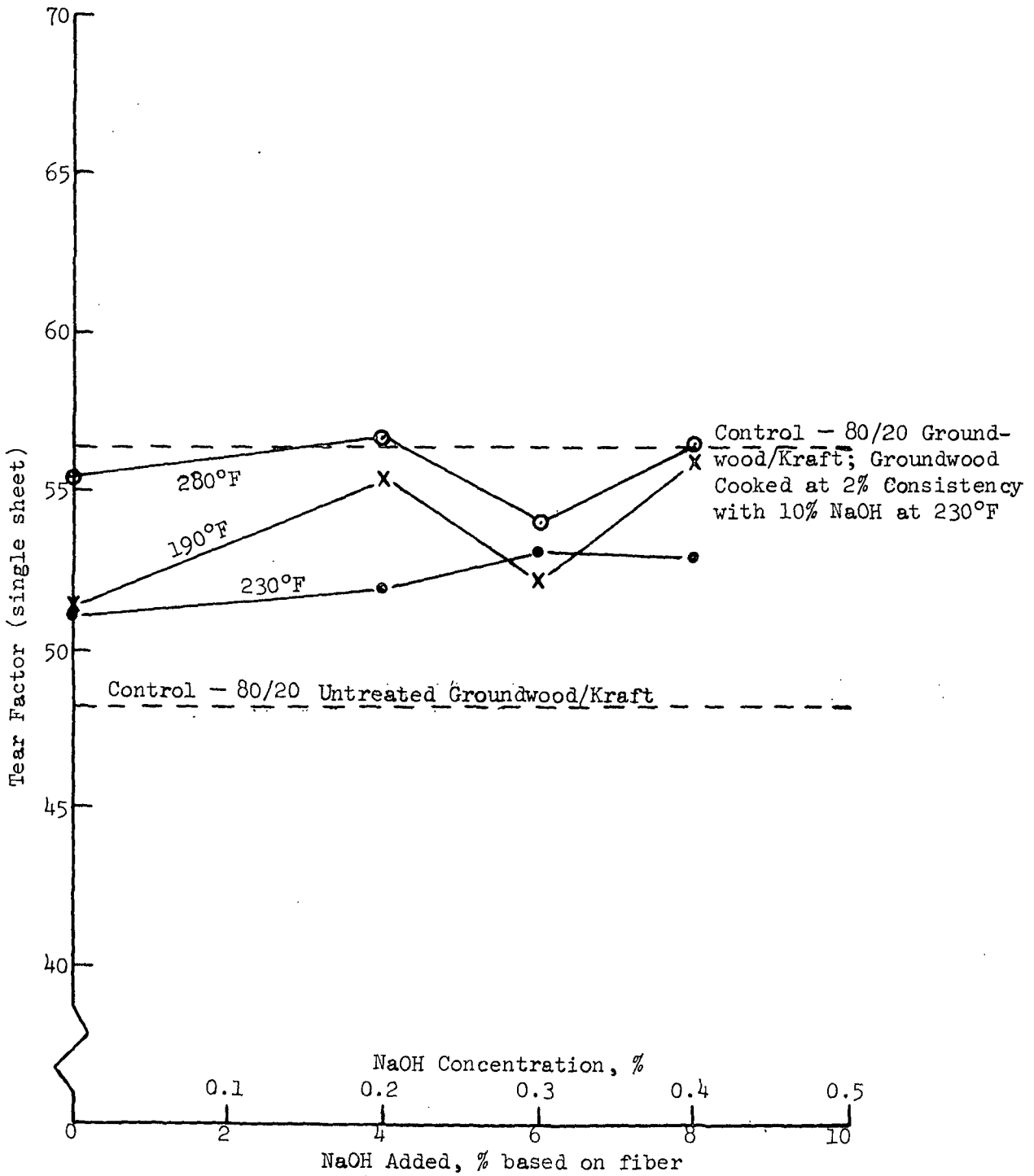

Figure 6. The Effect of Alkali Level and Temperature on Tear Factor (80/20 Blends of Groundwood/Kraft; Groundwood Cooked at 5% Consistency; 6-Sec Dwell Time)

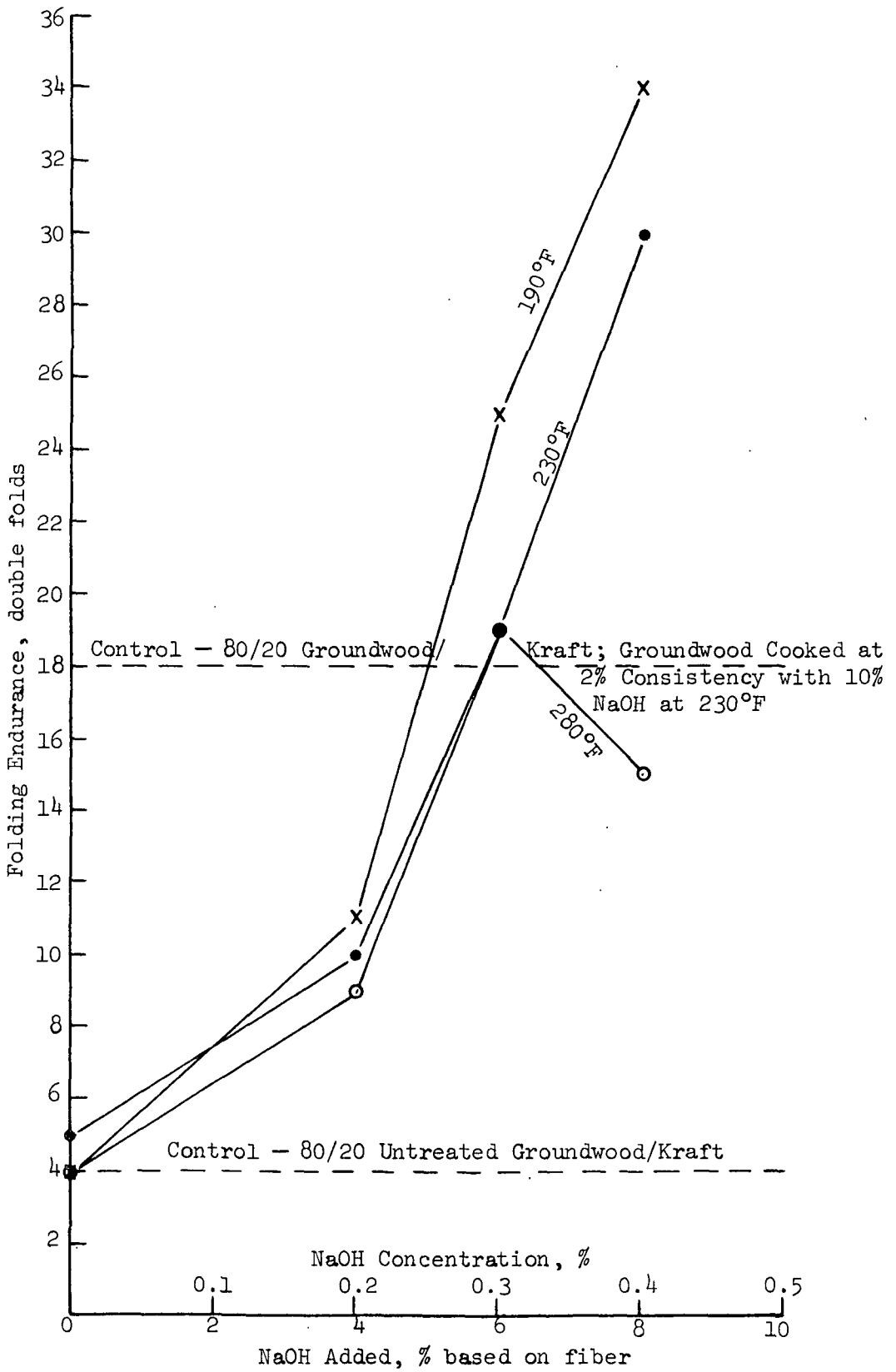

Figure 7. The Effect of Alkali Level and Temperature on Folding Endurance (80/20 Blends of Groundwood/Kraft; Groundwood Cooked at 5% Consistency; 6-Sec Dwell Time)

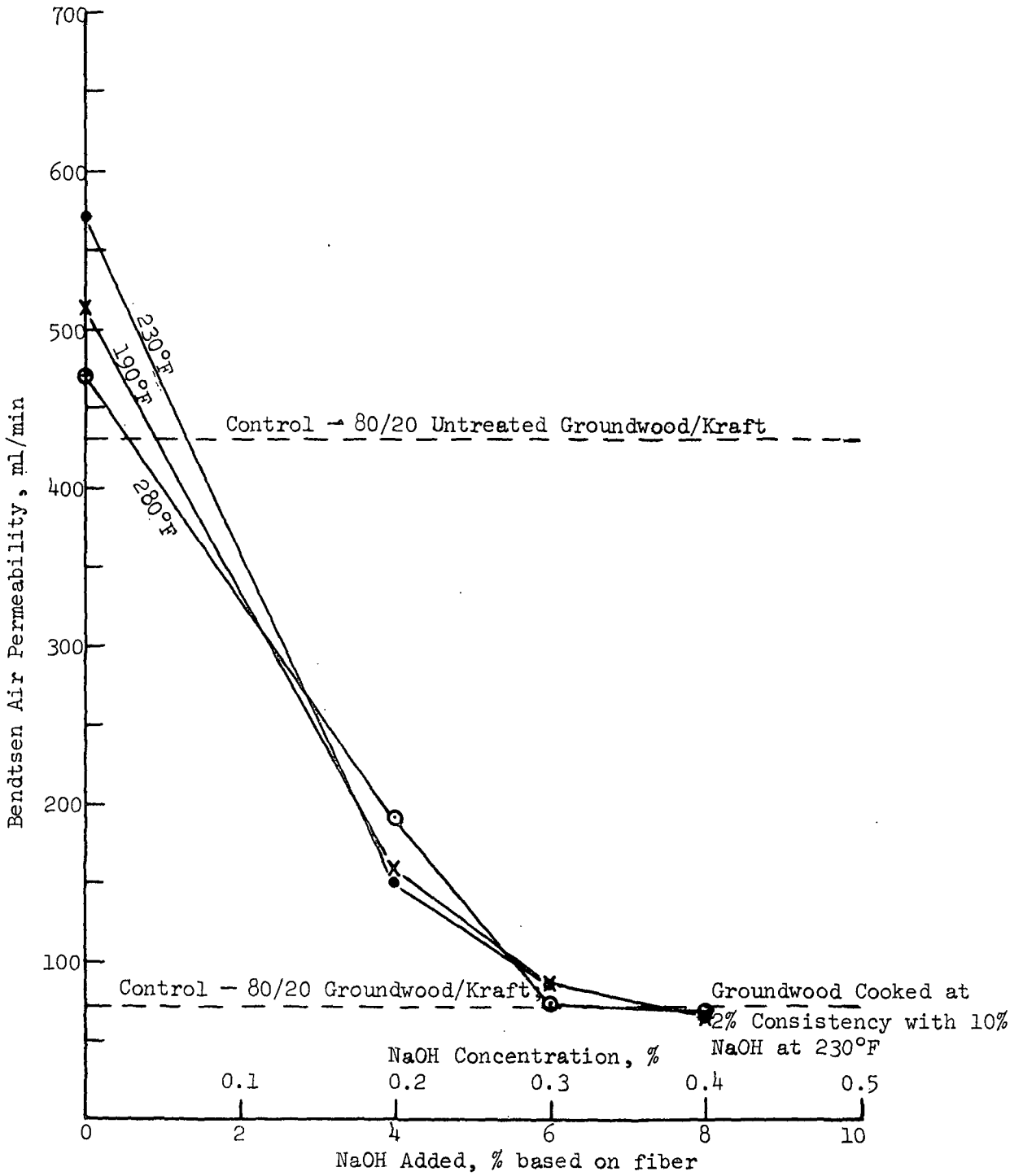

Figure 8. The Effect of Alkali Level and Temperature on Bendtsen Air Permeability (80/20 Blends of Groundwood/Kraft; Groundwood Cooked at 5% Consistency; 6-Sec Dwell Time)

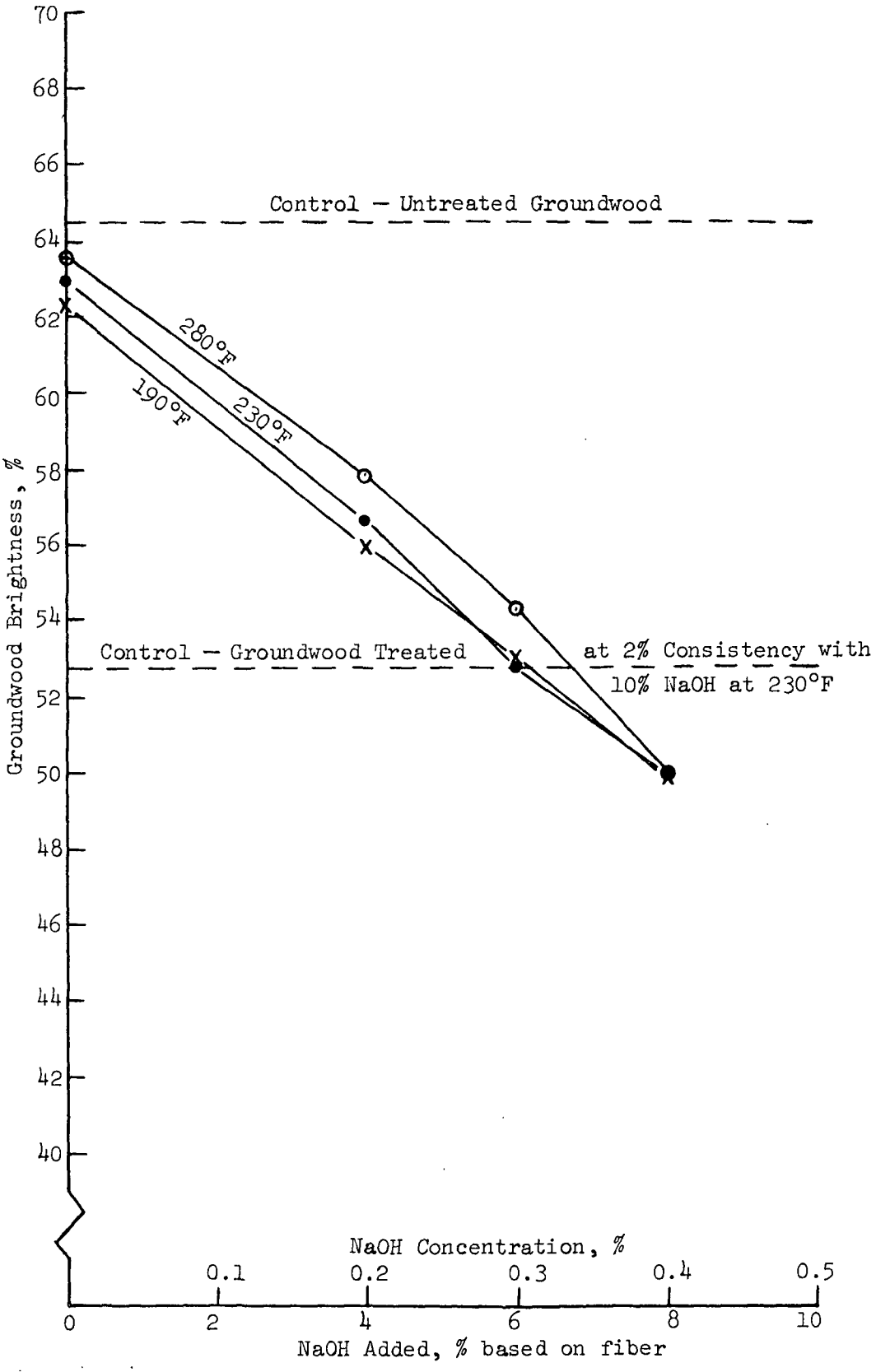

Figure 9. The Effect of Alkali Level and Temperature on the Brightness of Jet Cooked Groundwood (5% Consistency; 6-Sec Dwell Time)

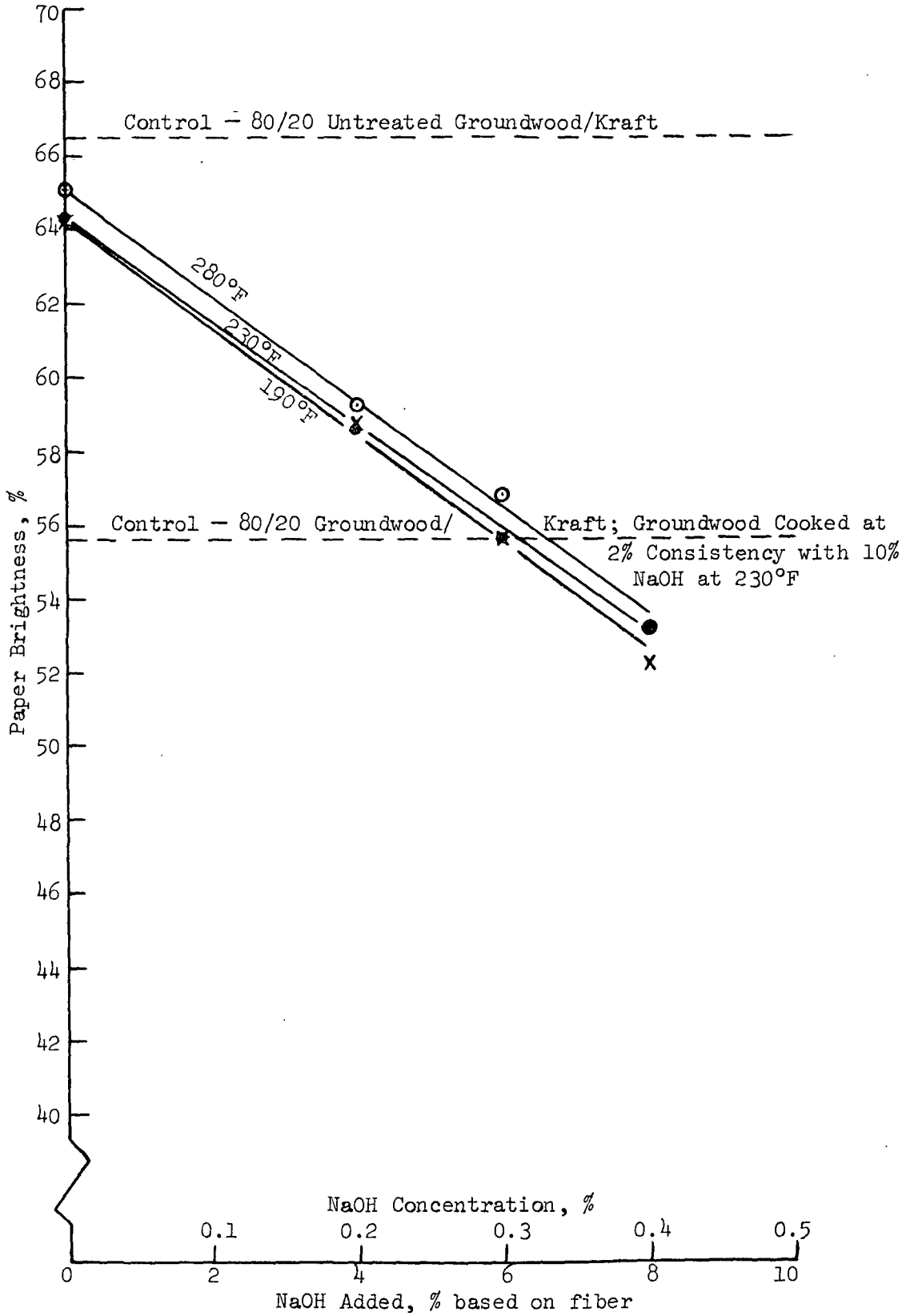

Figure 10. The Effect of Alkali Level and Temperature on Brightness (80/20 Blends of Groundwood/Kraft; Groundwood Cooked at 5% Consistency; 6-Sec Dwell Time)

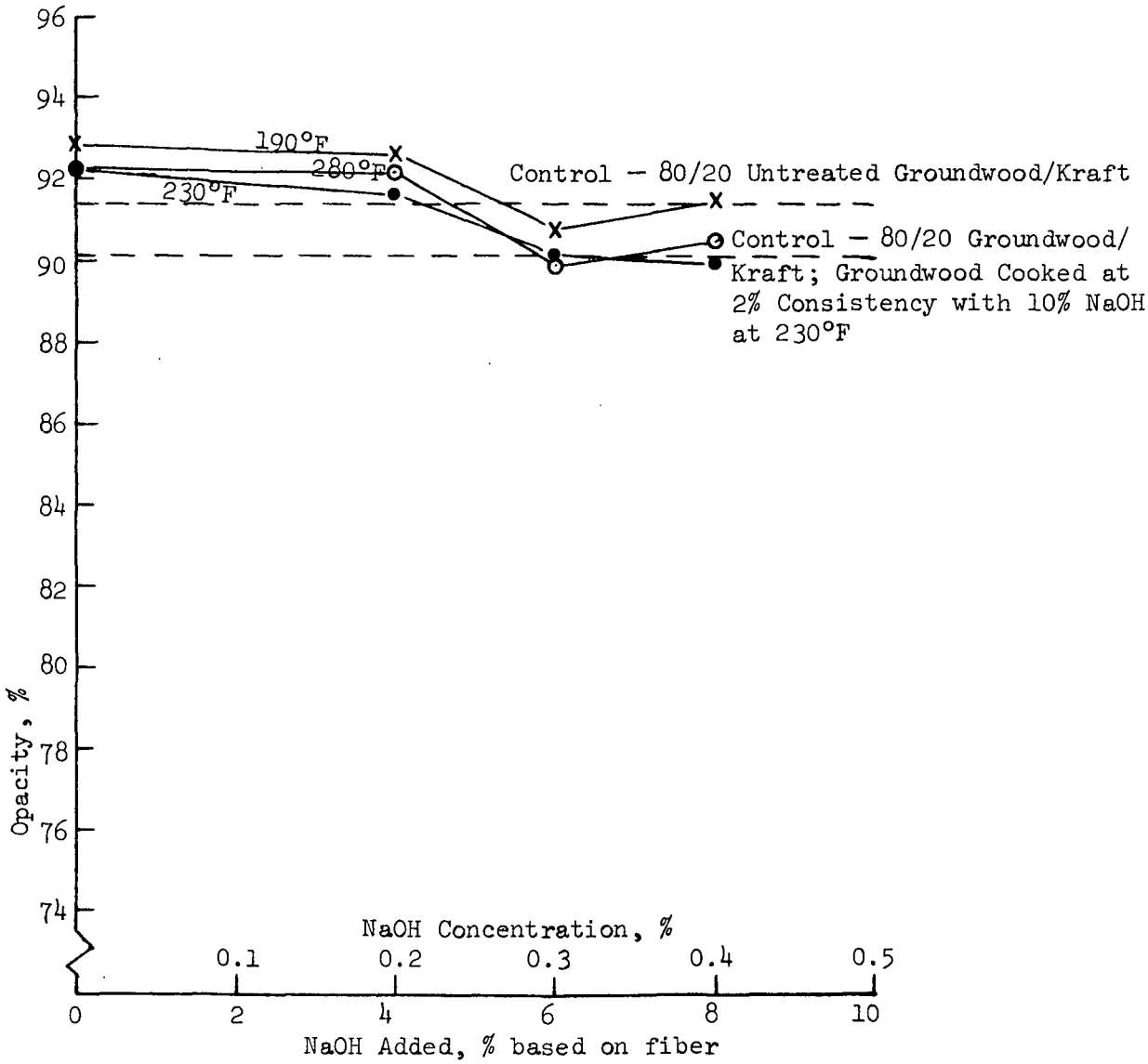

Figure 11. The Effect of Alkali Level and Temperature on Opacity (80/20 Blends of Groundwood/Kraft; Groundwood Cooked at 5% Consistency; 6-Sec Dwell Time)

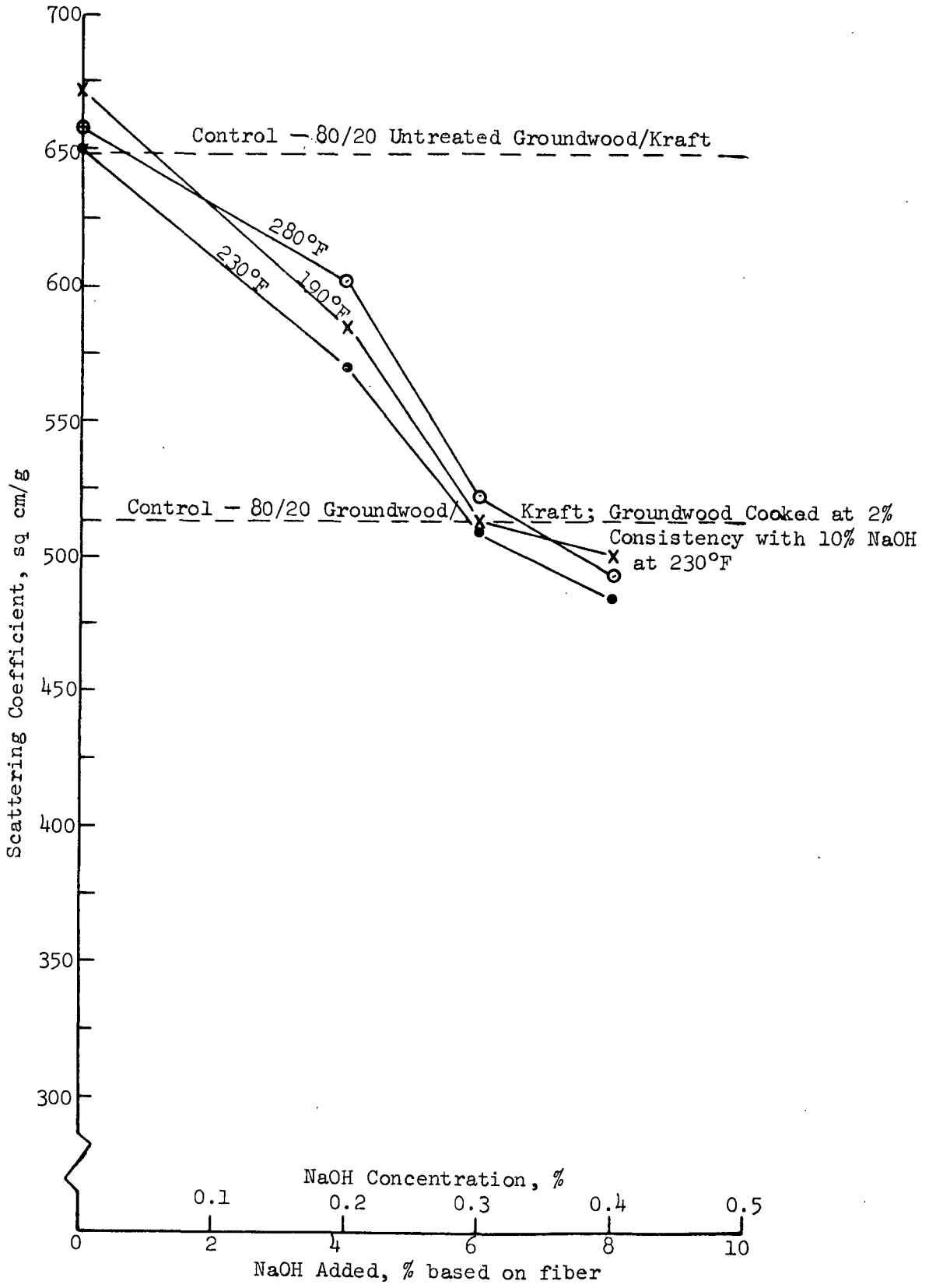

Figure 12. The Effect of Alkali Level and Temperature on Scattering Coefficient (80/20 Blends of Groundwood/Kraft; Groundwood Cooked at 5% Consistency; 6-Sec Dwell Time)

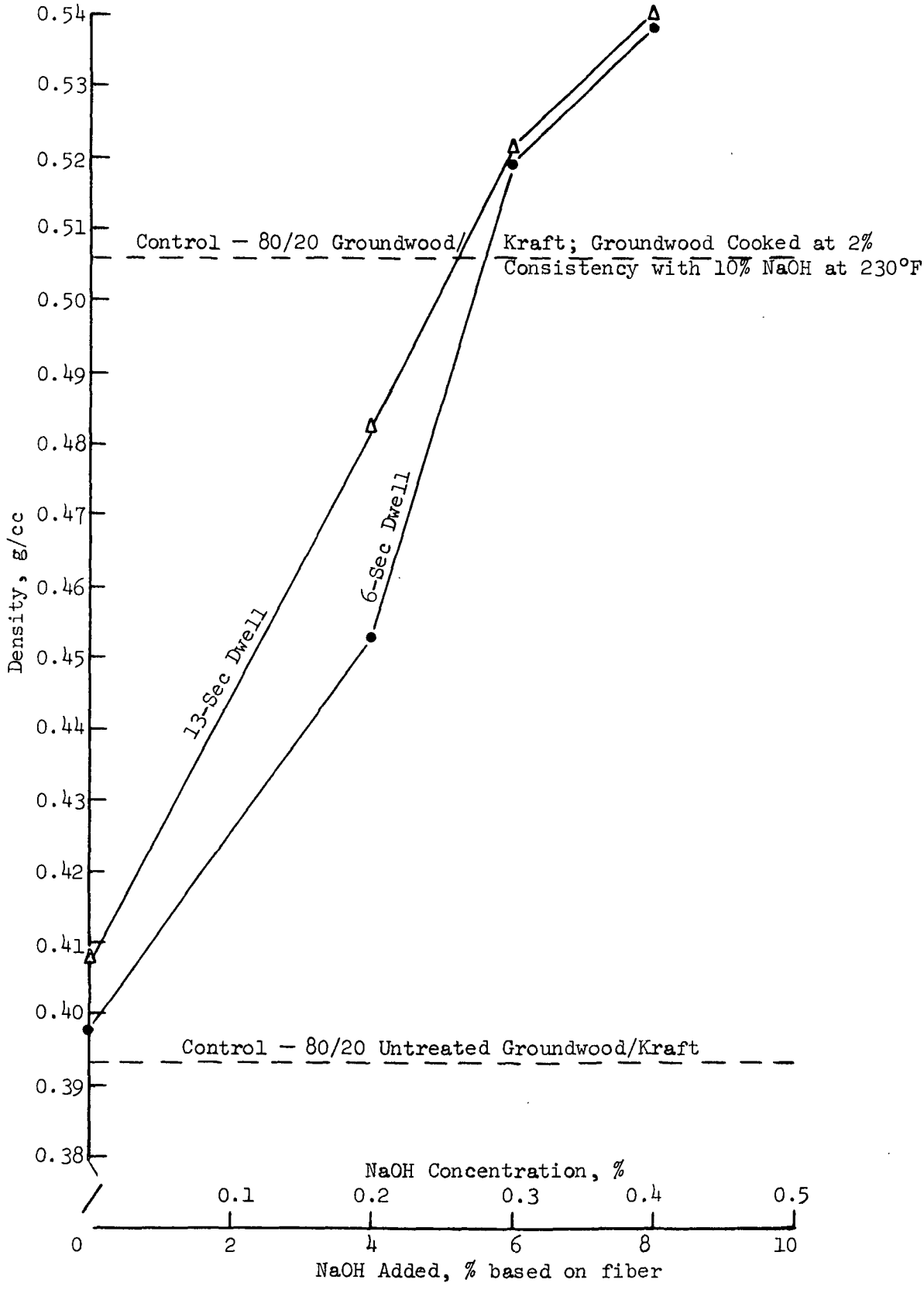

Figure 13. The Effect of Alkali Level and Dwell Time on Sheet Density (80/20 Blends of Groundwood/Kraft; Groundwood Cooked at 5% Consistency and 230°F)

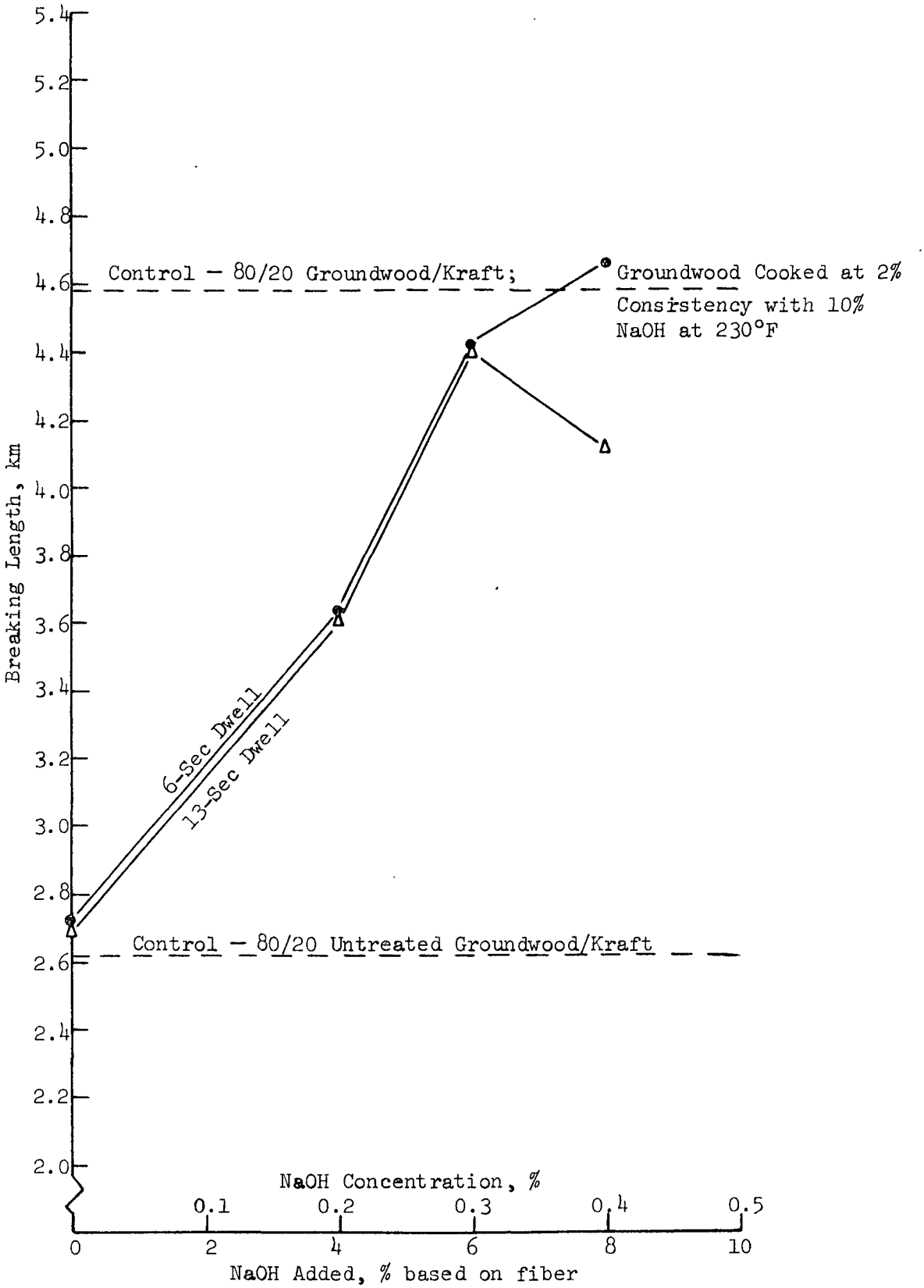

Figure 14. The Effect of Alkali Level and Dwell Time on Breaking Length (80/20 Blends of Groundwood/Kraft; Groundwood Cooked at 5% Consistency and 230°F)

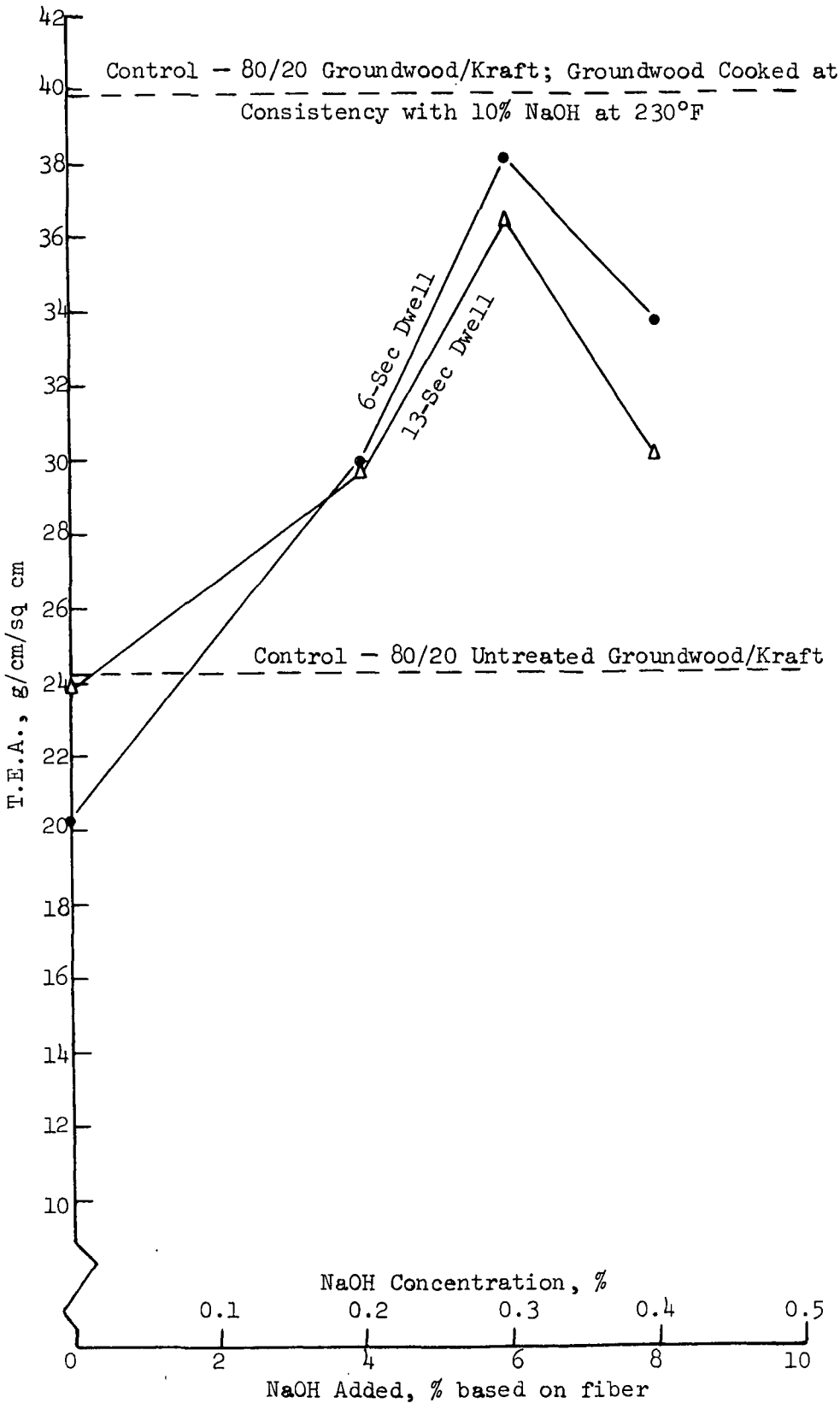

Figure 15. The Effect of Alkali Level and Dwell Time on Tensile Energy Absorption (80/20 Blends of Groundwood/Kraft; Groundwood Cooked at 5% Consistency and 230°F)

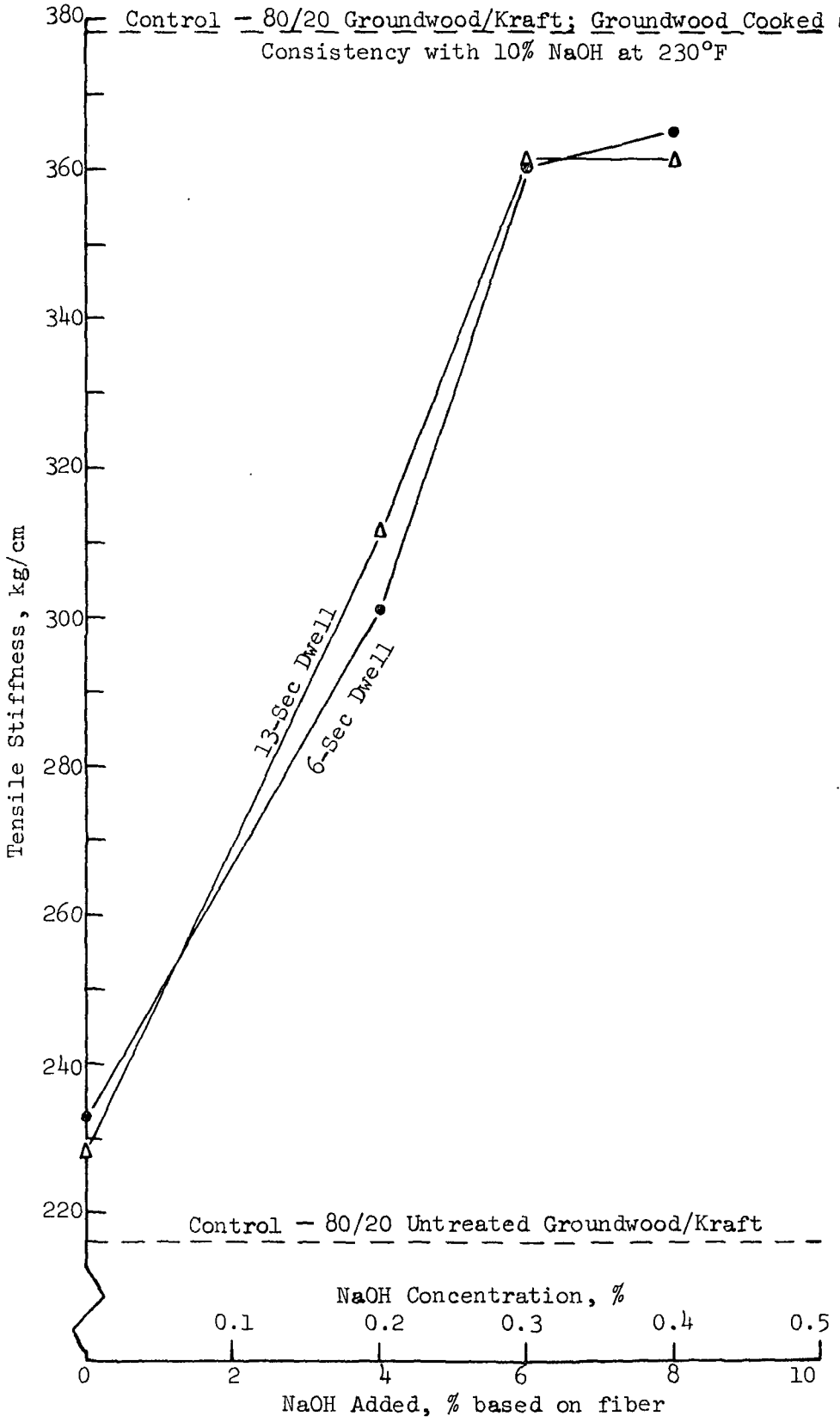

Figure 16. The Effect of Alkali Level and Dwell Time on Tensile Stiffness (80/20 Blends of Groundwood/Kraft; Groundwood Cooked at 5% Consistency and 230°F)

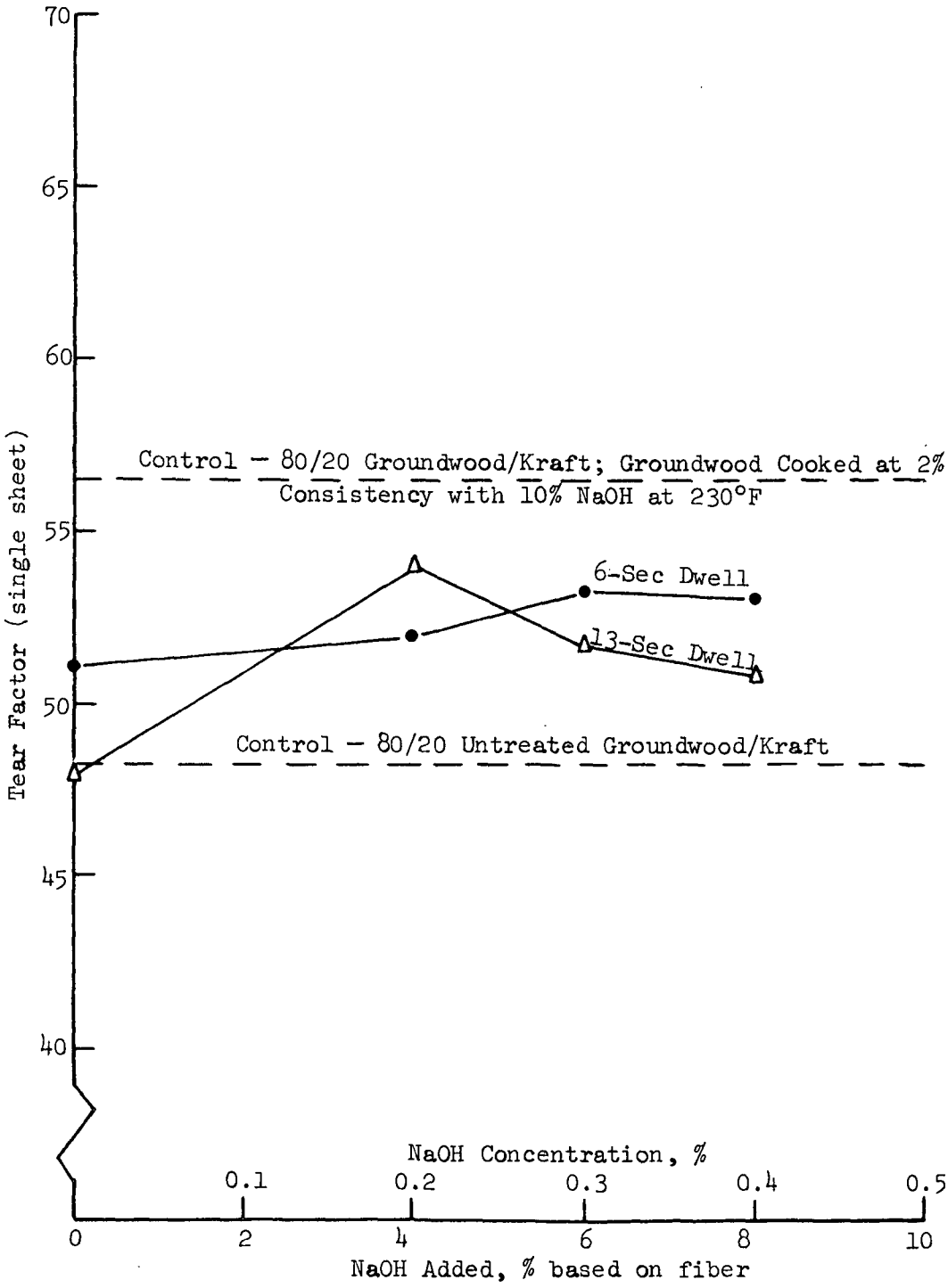

Figure 17. The Effect of Alkali Level and Dwell Time on Tear Factor (80/20 Blends of Groundwood/Kraft; Groundwood Cooked at 5% Consistency and 230°F)

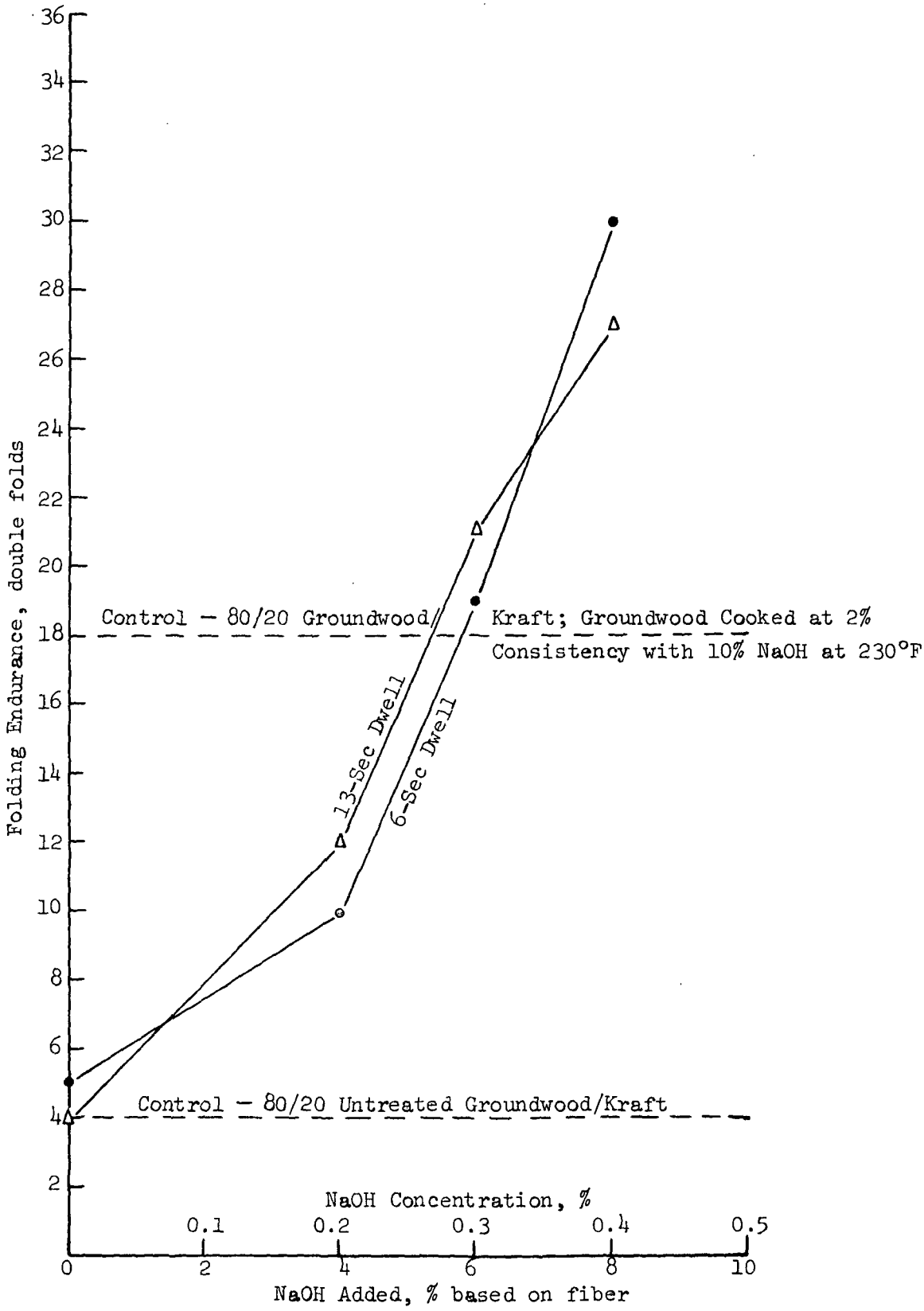

Figure 18. The Effect of Alkali Level and Dwell Time on Folding Endurance (80/20 Blends of Groundwood/Kraft; Groundwood Cooked at 5% Consistency and 230°F)

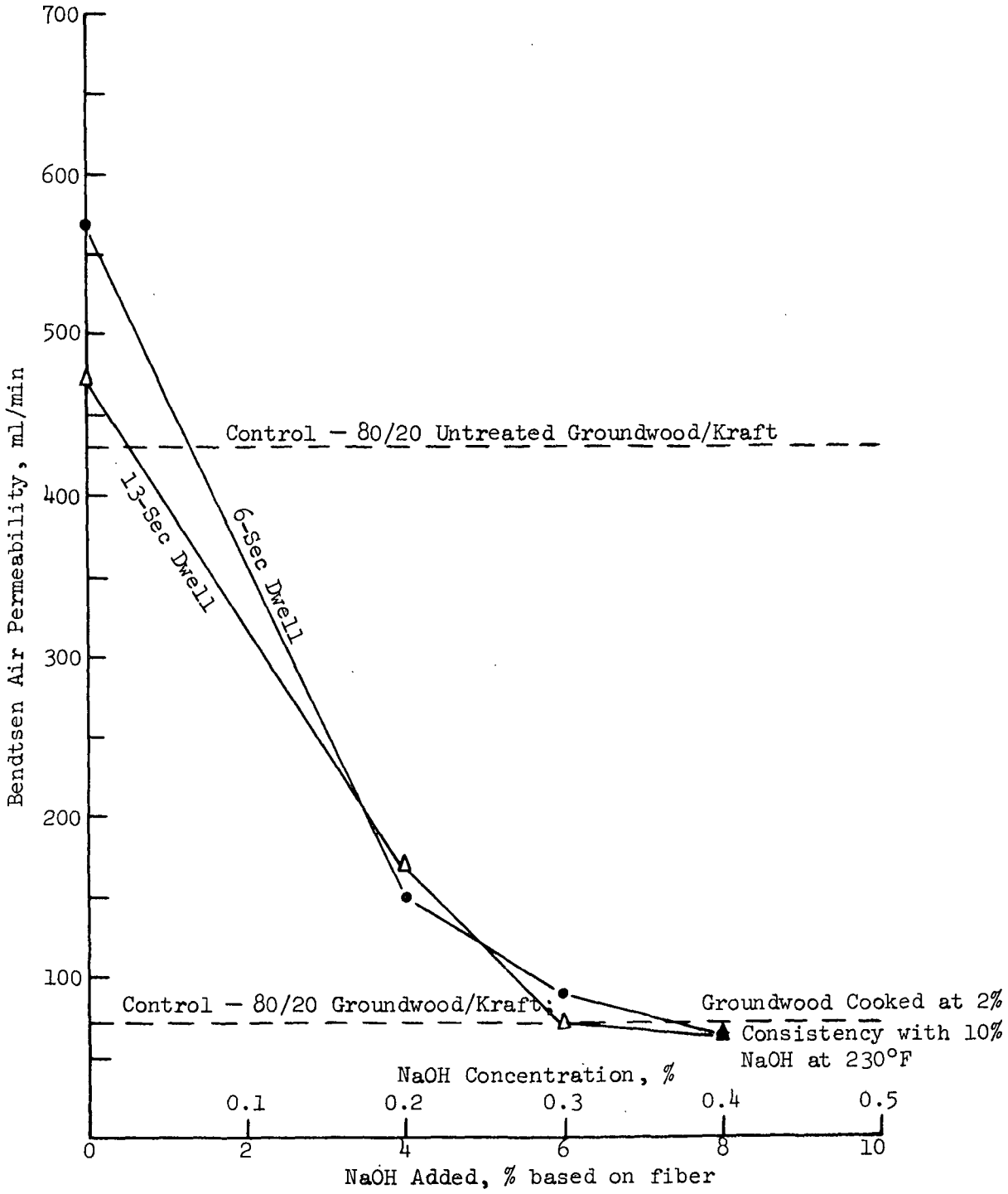

Figure 19. The Effect of Alkali Level and Dwell Time on Bendtsen Air Permeability (80/20 Blends of Groundwood/Kraft; Groundwood Cooked at 5% Consistency and 230°F)

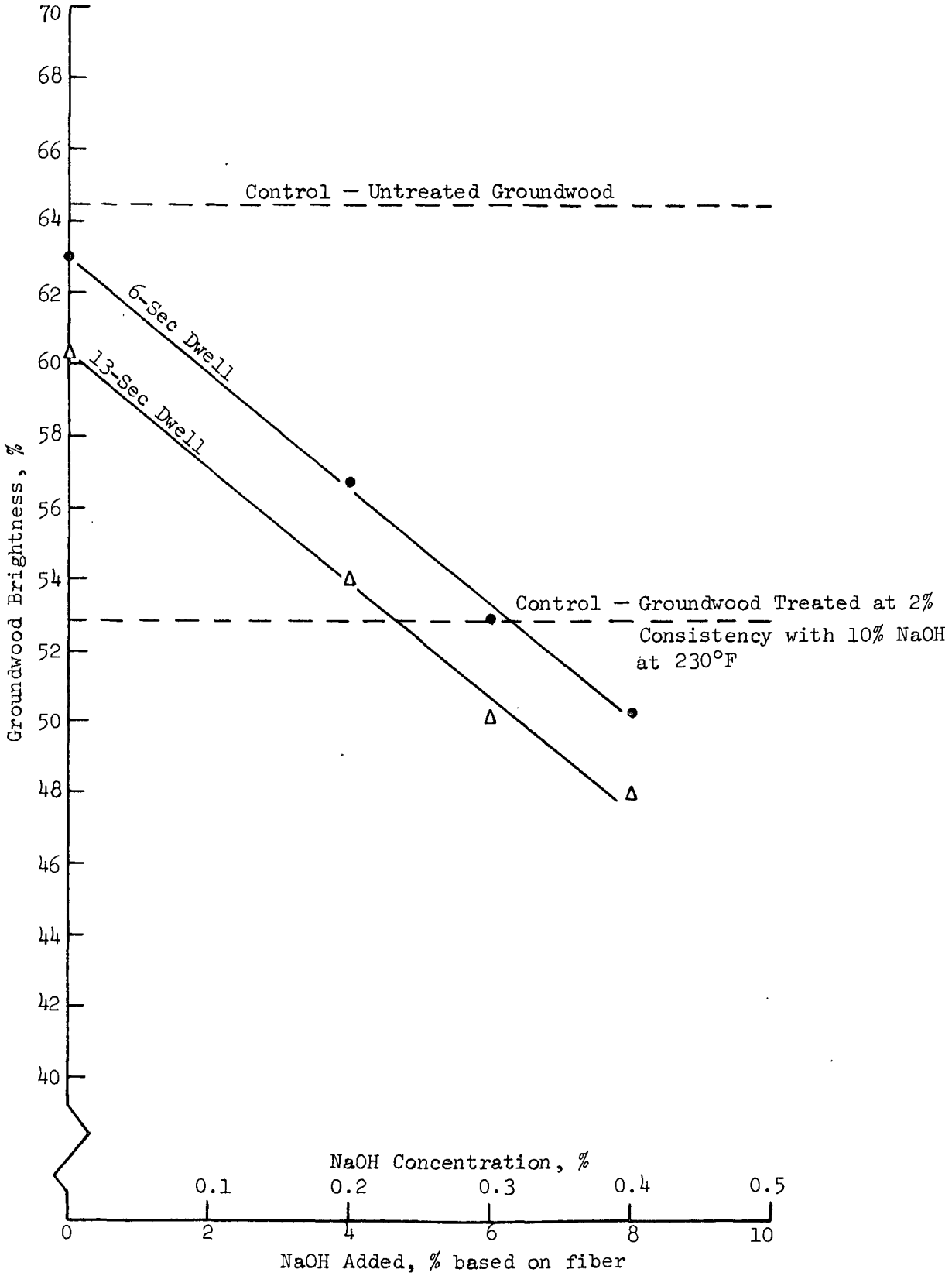

Figure 20. The Effect of Alkali Level and Dwell Time on the Brightness of Jet Cooked Groundwood (5% Consistency, 230°F)

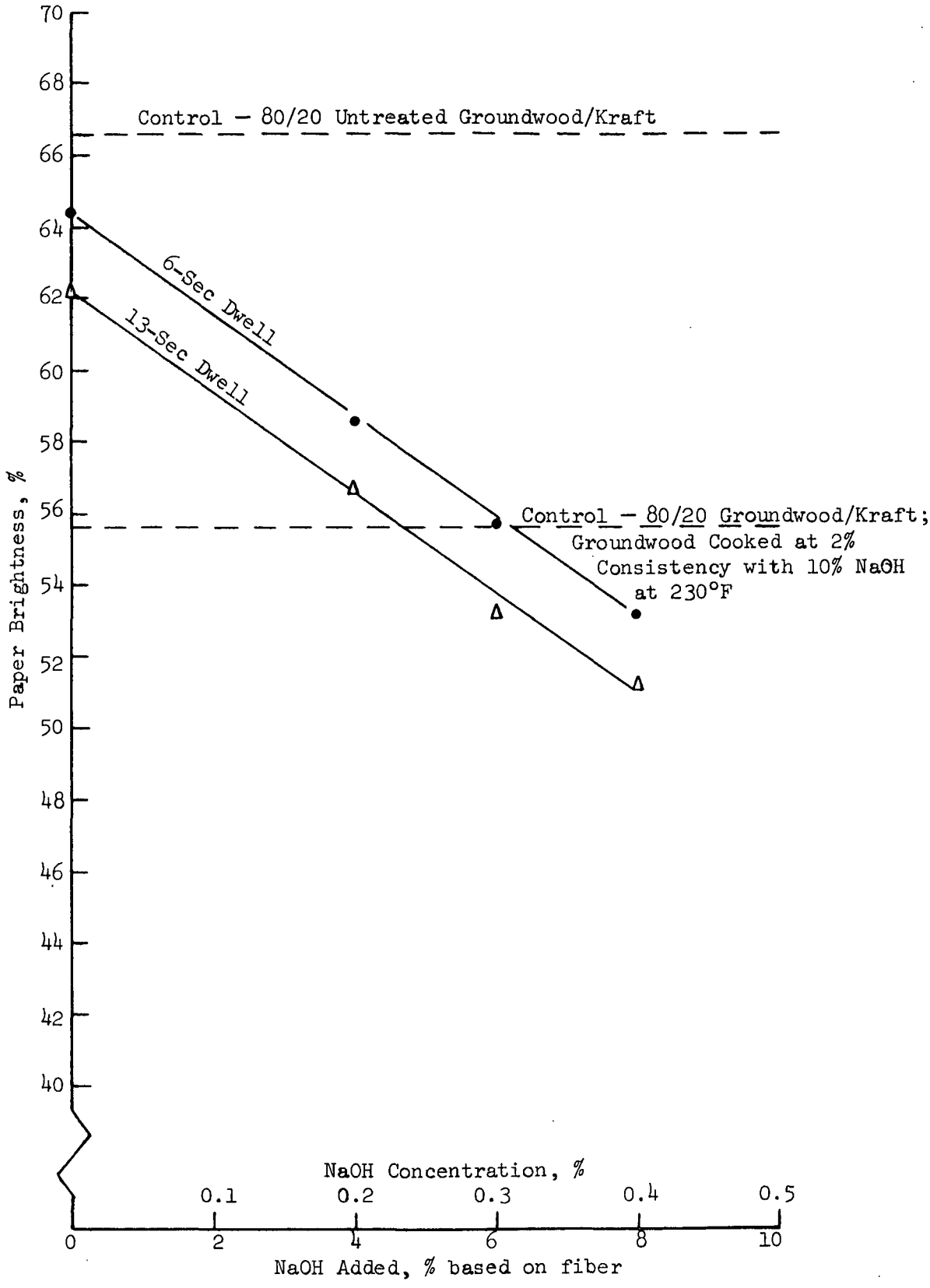

Figure 21. The Effect of Alkali Level and Dwell Time on Paper Brightness (80/20 Blends of Groundwood/Kraft; Groundwood Cooked at 5% Consistency and 230°F)

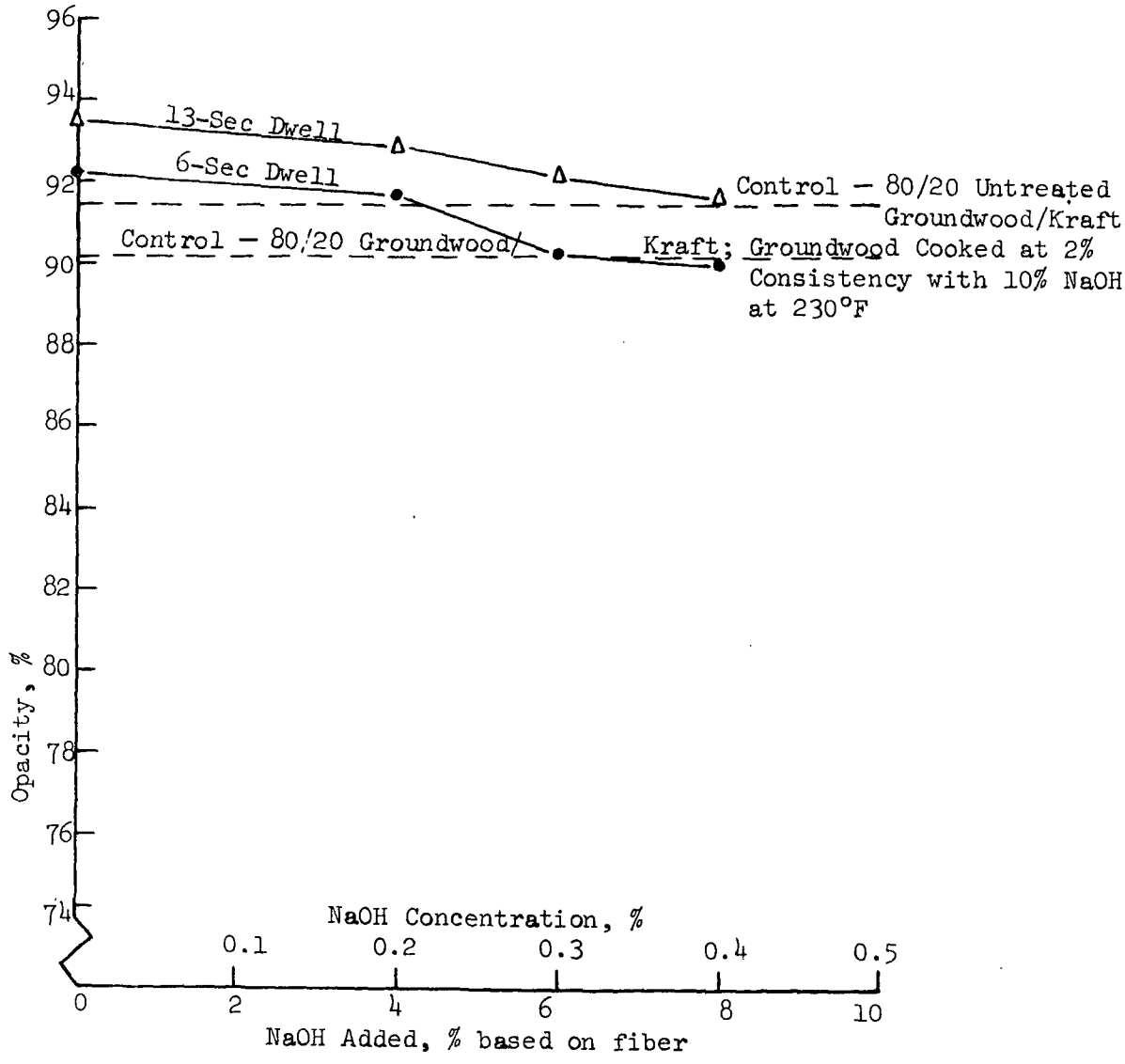

Figure 22. The Effect of Alkali Level and Dwell Time on Opacity (80/20 Blends of Groundwood/Kraft; Groundwood Cooked at 5% Consistency and 230°F)

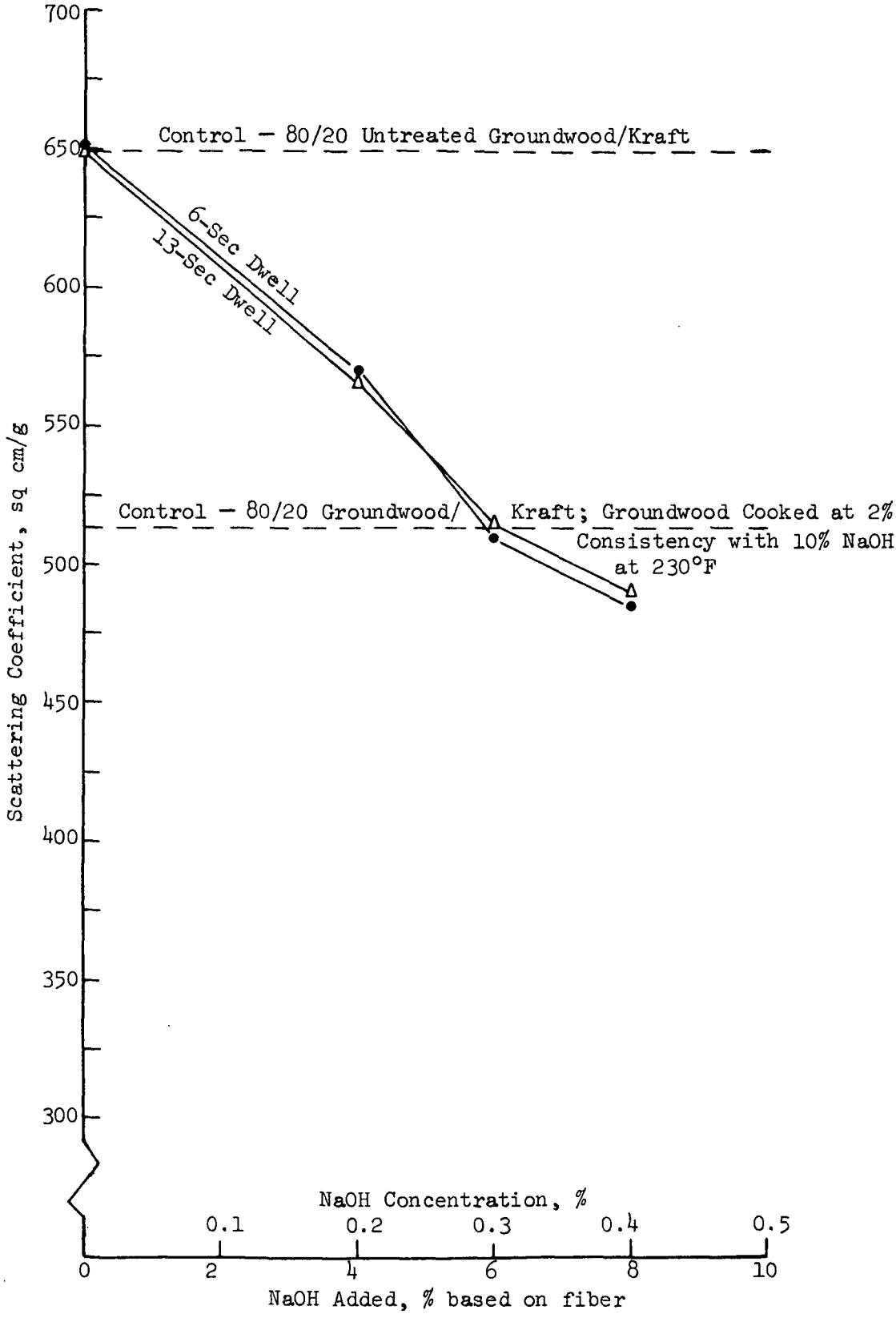

Figure 23. The Effect of Alkali Level and Dwell Time on Scattering Coefficient (80/20 Blends of Groundwood/Kraft; Groundwood Cooked at 5% Consistency and 230°F)

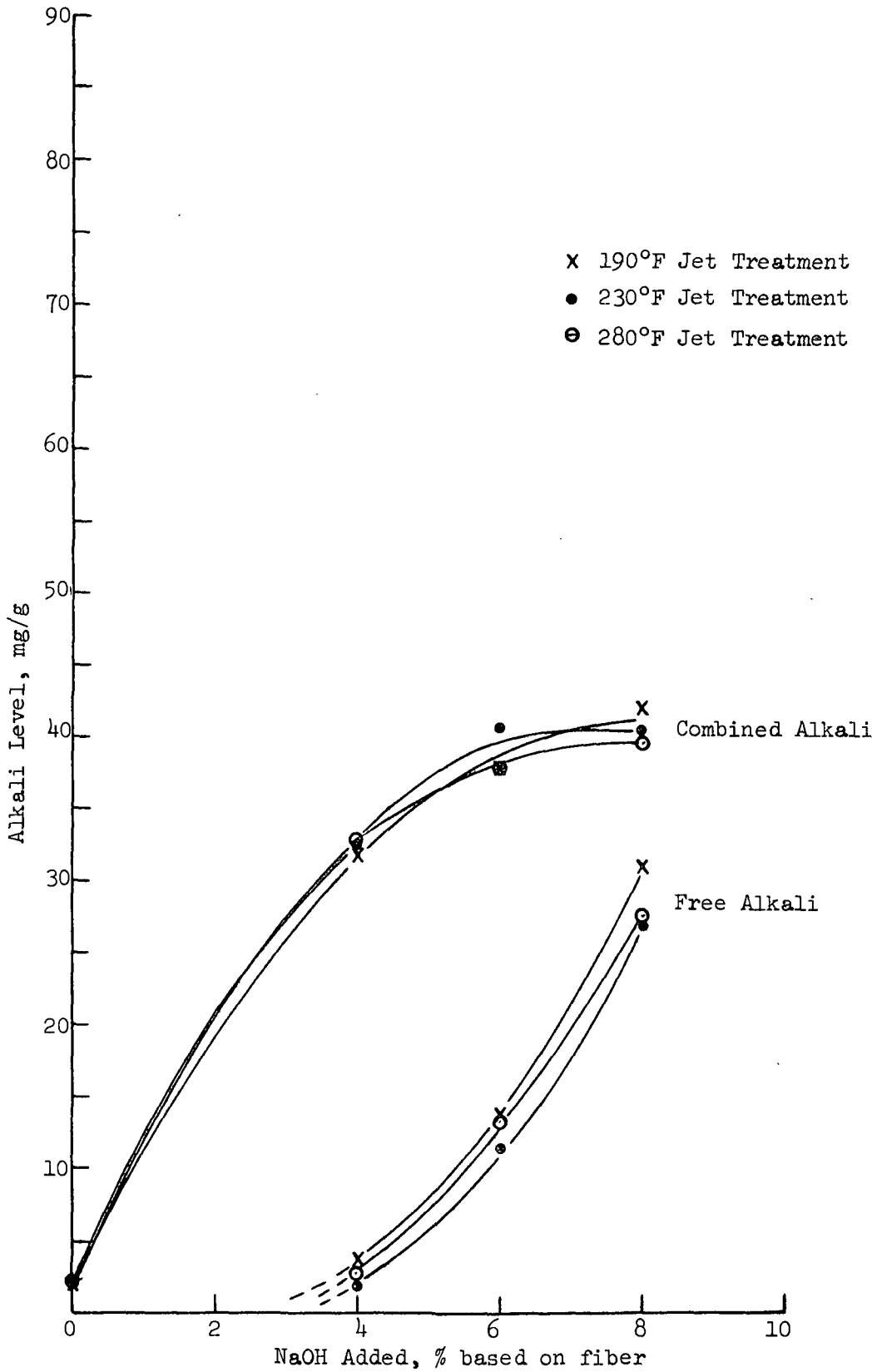

Figure 24. Free and Combined Alkali as a Function of Alkali Addition Level (6-Sec Dwell Time)

basis and in the form of Borol, a commercial product containing approximately 12% $NaBH_4$ and 42% NaOH. (Addition of 0.83 and 4.15% of Borol provided 0.1 and 0.5% of sodium borohydride.) The pH of the borohydride-treated pulp was reduced to 7 or to 5 with SO_2 after jet treatment. The jet treatments were initially made at a 6-sec dwell time and the more promising treatments were repeated at the 13-sec dwell time. Since the supply of groundwood had aged 5-6 months at this point in the program the decision was made to prepare fresh controls from 80/20 blends of untreated and jet/alkali-treated groundwood/kraft. Results are recorded in Tables V and VI. Brightness as a function of peroxide level is presented in Fig. 25.

Medium Density Bleaching Experiments

Medium density bleaching experiments were carried out on the following groundwood samples:

1. untreated
2. jet cooked 6 sec at 230°F with 6% NaOH
3. jet cooked with alkali as in No. 2 plus 1% of peroxide and 5% of sodium silicate

Samples 1 and 2 were bleached with 1 and 2% of H_2O_2, whereas Sample 3 was bleached with 1% of peroxide to provide the same total peroxide levels as in Samples 1 and 2. The experiments were carried out as follows:

Approximately 100-g samples of groundwood at 12.5% consistency were treated with the required amount of hydrogen peroxide, 5% of sodium silicate, and 0.05% of magnesium sulfate. The pulp samples were sealed in plastic bags and immersed in a water bath at 55°C. After two hours the samples were weighed and an aliquot was removed for yield determination.

TABLE V

THE EFFECT OF BRIGHTNESS LOSS INHIBITORS ON PHYSICAL PROPERTIES OF 80/20 BLENDS OF GROUNDWOOD AND KRAFT
(Groundwood component jet cooked at 5% consistency at 230°F with 6% of NaOH)

Set No.	Agents Added, % based on fiber	Dwell Time in Jet Cooker, sec	Basis Wt., g/M^2	S.E.	Thickness, µm	S.E.	Density, g/cc	Breaking Length, km	S.E.	T.E.A., g cm/sq cm	S.E.	Stretch, %	S.E.	Tensile Stiffness, kg/cm	S.E.
3-1[a]	Controls (80/20 untreated groundwood/kraft)	—	64.7	0.27	159.5	0.95	0.405	2.53	0.03	20.11	1.88	1.71	0.12	228	3.2
11-1[a]	Controls (80/20 jet-alkali-treated groundwood/kraft)	6	63.6	0.19	119.4	0.80	0.533	4.41	0.05	41.11	2.37	2.03	0.09	355	2.8
23	Controls (80/20 jet-alkali-treated groundwood/kraft)	13	63.3	0.10	121.4	0.51	0.521	4.40	0.04	36.35	1.18	1.86	0.05	361	5.1
25	Sodium sulfite, 1.0	6	63.9	0.24	117.9	0.62	0.542	4.63	0.07	42.08	1.61	2.00	0.07	373	8.4
26	Sodium sulfite, 3.0	6	63.0	0.15	116.3	0.95	0.542	4.77	0.06	45.67	0.69	2.11	0.02	361	9.9
27	Sodium sulfite, 7.0	6	63.1	0.18	120.9	1.30	0.521	4.46	0.04	36.01	0.60	1.82	0.02	364	6.5
38	Sodium sulfite, 3.0	13	62.3	0.57	118.4	0.62	0.526	4.84	0.07	43.77	1.84	2.06	0.05	363	9.6
28	Sodium silicate, 5; hydrogen peroxide, 1.0	6	62.4	0.13	125.5	1.52	0.498	4.29	0.05	36.93	1.35	1.93	0.06	334	7.6
32	Sodium silicate, 5; hydrogen peroxide, 2.0	6	63.9	0.09	129.5	0.81	0.493	4.18	0.03	38.01	1.56	2.00	0.07	335	2.4
29	Sodium silicate, 5; hydrogen peroxide, 3.0	6	63.9	0.16	128.5	0.62	0.497	4.21	0.05	40.71	1.54	2.10	0.07	333	6.3
30	Sodium silicate, 10; hydrogen peroxide, 1.0	6	62.9	0.16	126.5	0.95	0.497	4.26	0.06	37.37	1.71	1.92	0.06	344	3.2
31	Sodium silicate, 10; hydrogen peroxide, 3.0	6	63.6	0.25	132.6	2.03	0.480	4.02	0.06	34.81	0.89	1.90	0.04	327	9.1
39	Sodium silicate, 5; hydrogen peroxide, 1.0	13	62.1	0.15	125.0	1.24	0.497	4.19	0.05	31.97	1.42	1.79	0.07	348	8.3
33	Sodium borohydride (as is), 0.1 + SO_2 to pH 7	6	64.4	0.15	118.9	0.51	0.542	4.35	0.04	40.97	1.81	2.02	0.06	362	4.0
34	Sodium borohydride (as Borol), 0.1 + SO_2 to pH 7	6	63.4	0.20	119.9	0.51	0.529	4.60	0.08	42.19	2.26	2.02	0.07	368	5.3
35	Sodium borohydride (as is), 0.5 + SO_2 to pH 7[b]	6	62.8	0.17	116.8	1.14	0.538	4.38	0.07	33.73	2.23	1.73	0.08	368	1.8
36	Sodium borohydride (as Borol), 0.5 + SO_2 to pH 7[b]	6	62.7	0.24	115.8	0.62	0.542	4.57	0.05	42.44	1.34	2.06	0.05	363	5.8
37	Sodium borohydride (as Borol), 0.5 + SO_2 to pH 5[b]	6	63.3	0.31	116.3	0.95	0.544	4.89	0.13	42.81	2.60	1.94	0.07	400	10.2
40	Sodium borohydride (as Borol), 0.5 + SO_2 to pH 7[b]	13	64.2	0.26	117.9	0.62	0.545	4.69	0.11	41.76	3.23	1.94	0.07	385	4.7
41	Sodium borohydride (as Borol), 0.5 + SO_2 to pH 5[b]	13	62.7	0.11	114.8	0.51	0.546	4.71	0.13	39.20	4.59	1.87	0.15	371	5.5

See end of table for footnotes.

TABLE V (Continued)

THE EFFECT OF BRIGHTNESS LOSS INHIBITORS ON PHYSICAL PROPERTIES OF 80/20 BLENDS OF GROUNDWOOD AND KRAFT
(Groundwood component jet cooked at 5% consistency at 230°F with 6% of NaOH)

Set No.	Agents Added, % based on fiber	Dwell Time in Jet Cooker, sec	Tear Factor, (single sheet)	S.E.	MIT Folding Endurance, double folds	S.E.	Bendtsen Air Permeability, ml/min	S.E.	Groundwood Brightness, %	S.E.	Paper Brightness, %	S.E.	Opacity, %	S.E.	Scattering Coefficient, sq cm/g	S.E.
3-1[a]	Controls (80/20 untreated groundwood/kraft)	--	49.2	1.7	3	0.2	439	8.3	63.6	0.14	64.8	0.10	92.5	0.09	661	3.8
11-1[a]	Controls (80/20 jet-alkali-treated groundwood/kraft)	6	51.9	1.7	21	1.3	71	1.9	50.8	0.07	53.4	0.20	91.5	0.26	509	6.9
23	Controls (80/20 jet-alkali-treated groundwood/kraft)	13	51.8	1.3	21	1.0	72	1.6	50.1	0.24	53.2	0.35	92.1	0.10	515	5.0
25	Sodium sulfite, 1.0	6	53.5	1.0	26	1.3	63	0.8	52.0	0.27	53.9	0.37	91.0	0.35	489	7.9
26	Sodium sulfite, 3.0	6	52.0	0.9	22	1.4	63	2.2	54.7	0.10	57.0	0.13	90.3	0.10	504	3.8
27	Sodium sulfite, 7.0	6	49.2	1.4	24	1.5	71	2.2	55.4	0.16	57.6	0.38	90.1	0.26	504	4.6
38	Sodium sulfite, 3.0	13	44.0	4.1	30	2.7	71	2.1	54.5	0.19	57.7	0.09	90.6	0.11	515	1.6
28	Sodium silicate, 5; hydrogen peroxide, 1.0	6	56.7	5.1	17	1.1	93	2.8	60.7	0.19	63.1	0.15	87.7	0.07	524	1.2
32	Sodium silicate, 5; hydrogen peroxide, 2.0	6	49.2	1.2	17	0.8	110	2.5	67.9	0.22	69.6	0.11	87.1	0.12	545	3.4
29	Sodium silicate, 5; hydrogen peroxide, 3.0	6	53.8	1.2	15	0.8	112	1.3	69.7	0.15	70.8	0.12	87.5	0.14	561	4.5
30	Sodium silicate, 10; hydrogen peroxide, 1.0	6	52.2	1.3	14	0.7	106	2.1	59.9	0.23	62.4	0.14	89.1	0.19	549	5.7
31	Sodium silicate, 10; hydrogen peroxide, 3.0	6	51.9	1.0	13	0.7	126	4.2	70.9	0.36	71.2	0.27	87.7	0.26	566	6.8
39	Sodium silicate, 5; hydrogen peroxide, 1.0	13	55.7	1.6	15	1.2	130	7.2	60.4	0.17	62.2	0.10	88.6	0.16	532	3.5
33	Sodium borohydride (as is), 0.1 + SO_2 to pH 7	6	53.4	1.5	15	1.0	67	1.3	53.0	0.15	55.9	0.20	90.2	0.18	503	4.4
34	Sodium borohydride (as Borol), 0.1 + SO_2 to pH 7	6	53.3	2.7	23	1.2	66	4.2	53.1	0.26	55.8	0.11	89.8	0.34	500	8.8
35	Sodium borohydride (as is), 0.5 + SO_2 to pH 7[b]	6	50.6	1.1	19	1.3	62	3.3	58.7	0.22	61.2	0.27	88.7	0.24	500	5.6
36	Sodium borohydride (as Borol), 0.5 + SO_2 to pH 7[b]	6	51.0	1.1	20	1.1	55	3.2	58.2	0.45	60.5	0.13	88.9	0.31	501	7.0
37	Sodium borohydride (as Borol), 0.5 + SO_2 to pH 5[b]	6	56.3	1.8	28	2.2	52	0.7	56.1	0.35	59.5	0.24	89.3	0.32	496	9.8
40	Sodium borohydride (as Borol), 0.5 + SO_2 to pH 7[b]	13	53.0	0.8	30	2.6	72	4.2	55.8	0.16	58.9	0.24	88.8	0.30	472	5.7
41	Sodium borohydride (as Borol), 0.5 + SO_2 to pH 5[b]	13	54.9	2.5	33	3.4	56	1.4	56.0	0.36	58.2	0.38	89.9	0.43	487	5.7

[a] These control sets represent repeat preparations of Sets 3 and 11 at a later date than the original sets.
[b] pH adjusted with SO_2 after jet cooking.

Note: The set numbers reflect the order in which the sets were made.
0.1% of sodium borohydride is equivalent to 0.83% of Borol.
0.5% of sodium borohydride is equivalent to 4.15% of Borol.

TABLE VI

GROUNDWOOD SUSPENSION DATA — BRIGHTNESS LOSS SERIES
(Groundwood jet cooked at 5% consistency at 230°F with 6% of NaOH)

Cook No.	Agents Added, % based on fiber	Dwell Time, sec	pH of Pulp Slurry		Total Solids in Pulp Slurry, %	Sol. Solids in Pulp Slurry, %	Approx. Yield,[a] %	Residual H_2O_2	
			Before Cook	After Cook				Mg/g	% Left
20	Sodium sulfite, 1.0	6	12.4	12.1	4.84	0.59	96.3	--	--
21	Sodium sulfite, 3.0	6	12.5	12.3	4.82	0.73	94.7	--	--
22	Sodium sulfite, 7.0	6	12.5	12.3	4.95	0.84	95.7	--	--
23	Sodium silicate, 5.0; hydrogen peroxide, 1.0	6	12.2	11.6	4.74	0.63	98.5	0.84	9.3
24	Sodium silicate, 5.0; hydrogen peroxide, 3.0	6	11.9	10.9	4.66	0.64	98.0	13.96	51.7
25	Sodium silicate, 10.0; hydrogen peroxide, 1.0	6	12.2	11.4	4.70	0.66	100+	1.70	19.7
26	Sodium silicate, 10.0; hydrogen peroxide, 3.0	6	11.7	10.9	4.79	0.71	100+	12.1	46.9
27	Sodium silicate, 5.0; hydrogen peroxide, 2.0	6	12.0	11.1	4.83	0.68	97.6	7.25	40.3
28	Sodium borohydride (as is), 0.1; SO_2 to pH 7	6	12.6	12.2	4.66	0.58	95.4	--	--
29	Sodium borohydride (as Borol), 0.1; SO_2 to pH 7	6	12.6	12.4	4.71	0.65	93.9	--	--
30	Sodium borohydride (as is), 0.5; SO_2 to pH 7	6	12.6	12.3	4.65	0.64	94.5	--	--
31	Sodium borohydride (as Borol), 0.5; SO_2 to pH 7	6	12.8	12.7	4.76	0.79	91.4	--	--
32	Sodium borohydride (as Borol), 0.5; SO_2 to pH 5	6	12.7	12.6	4.92	0.79	92.0	--	--
33	Sodium sulfite, 3.0	13	12.6	12.3	4.86	0.71	95.3	--	--
34	Sodium silicate, 5.0; hydrogen peroxide, 1.0	13	12.0	11.1	4.80	0.60	99.4	1.25	13.9
35	Sodium borohydride (as Borol), 0.5; SO_2 to pH 7	13	12.6	12.6	5.07	0.77	92.9	--	--
36	Sodium borohydride (as Borol), 0.5; SO_2 to pH 5	13	12.6	12.6	4.98	0.76	92.8	--	--

[a]The yield values listed above are considered approximate.

Note: 0.1% of sodium borohydride is equivalent to 0.83% of Borol.
0.5% of sodium borohydride is equivalent to 4.15% of Borol.

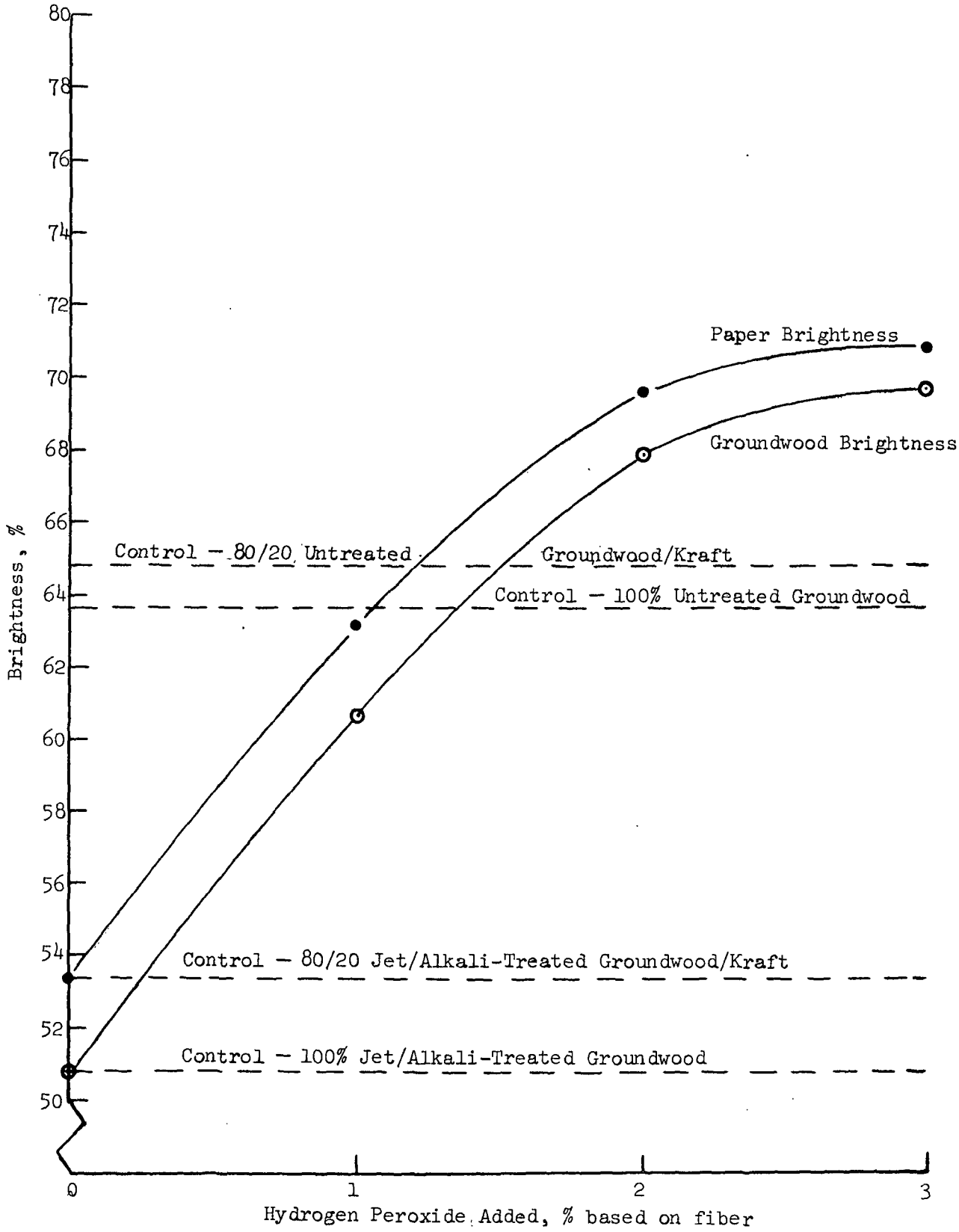

Figure 25. Brightness as a Function of Hydrogen Peroxide Level in the Jet/Alkali Cooking Liquor

Yield was determined from this aliquot by materials balance. The remaining portion of the original bleached pulp sample was diluted and the pH adjusted to 5.5 with sulfurous acid. This material was then dewatered on a singered glass funnel and subsequently used to prepare handsheets for strength, structural, and optical properties in the manner previously described. Results are recorded in Tables VII and VIII. Brightness as a function of peroxide level is plotted in Fig. 26 and 27.

Properties of All-Groundwood Pulps and Papers

Based on Forgacs' work (4), classification tests were conducted on selected samples of unbleached groundwood. In general, the pulps selected for this purpose were those which previously provided optimum or near optimum properties when blended with kraft. Ten-gram samples of these pulps were processed on the Bauer-McNett classifier according to TAPPI Method T 233 sw-64 but using only 28, 48, and 100-mesh screens. The fraction passing through the 48-mesh screen but retained on the 100 mesh was collected and weighed. An amount of this pulp equivalent to three grams was then used for Canadian freeness measurements. At least two 10-gram samples of pulp were required to provide the necessary amount for the freeness tests. According to Forgacs, the Canadian freeness of the 48/100 fraction is a measure of the specific surface and is related to the bonding potential of a mechanical pulp.

Handsheets previously formed for brightness tests from the same groundwood pulps utilized in the classification experiments were also tested for other strength, structural, and optical properties to examine the potential of 100% groundwood papers. Several samples of pulp from the medium density bleaching experiments were included in this series. Results for the all-groundwood papers, as well as those for the blended groundwood/kraft controls, are

TABLE VII

PHYSICAL PROPERTIES OF 80/20 BLEACHED GROUNDWOOD/KRAFT HANDSHEETS
(Medium density bleaching experiments)

Set No.	Groundwood Treatment Prior to Bleaching	H_2O_2 Added in Bleaching, % based on fiber	Basis Weight, g/M^2	S.E.	Thickness, µm	S.E.	Density, g/cc	Breaking Length, km	S.E.	T.E.A., g cm/sq cm	S.E.	Stretch, %	S.E.	Tensile Stiffness, kg/cm	S.E.
3-1	None, untreated groundwood (control)	0	64.7	0.27	159.5	0.95	0.405	2.53	0.03	20.11	1.88	1.71	0.12	228	3.2
42	None, untreated groundwood	1	62.9	0.22	161.5	0.63	0.390	2.71	0.03	20.95	1.18	1.76	0.08	188	4.5
43	None, untreated groundwood	2	63.2	0.23	159.0	1.02	0.397	2.63	0.03	21.11	1.81	1.83	0.12	174	3.5
11-1	Jet cooked 6 sec at 230°F with 6% NaOH	0	63.6	0.19	119.4	0.80	0.533	4.41	0.05	41.11	2.37	2.03	0.09	355	2.8
44	Jet cooked 6 sec at 230°F with 6% NaOH	1	62.8	0.16	117.9	1.30	0.533	4.65	0.04	43.15	3.26	2.06	0.12	292	8.6
45	Jet cooked 6 sec at 230°F with 6% NaOH	2	63.0	0.07	115.8	0.62	0.544	4.84	0.05	47.03	2.64	2.15	0.10	296	1.9
28	Jet cooked 6 sec at 230°F with 6% NaOH, 5% Na_2SiO_3, & 1% H_2O_2	0	62.4	0.13	125.5	1.52	0.498	4.29	0.05	36.93	1.35	1.93	0.06	334	7.6
46	Jet cooked 6 sec at 230°F with 6% NaOH, 5% Na_2SiO_3, & 1% H_2O_2	1	63.5	0.17	118.9	0.95	0.534	4.70	0.16	38.48	3.17	1.83	0.10	386	11.6

Set No.	Groundwood Treatment Prior to Bleaching	H_2O_2 Added in Bleaching, % based on fiber	Tear Factor, (single sheet)	S.E.	MIT Folding Endurance, double folds	S.E.	Bendtsen Air Permeability, ml/min	S.E.	Groundwood Brightness, %	S.E.	Paper Brightness, %	S.E.	Opacity, %	S.E.	Scattering Coeff., sq cm/g	S.E.
3-1	None, untreated groundwood (control)	0	49.2	1.7	3	0.2	439	8.3	63.6	0.14	64.8	0.10	92.5	0.09	661	3.8
42	None, untreated groundwood	1	51.5	1.2	6	0.3	440	15.2	71.1	0.06	72.0	0.06	89.6	0.11	647	5.2
43	None, untreated groundwood	2	51.6	1.0	5	0.3	567	7.8	73.0	0.14	73.8	0.11	89.1	0.09	646	3.1
11-1	Jet cooked 6 sec at 230°F with 6% NaOH	0	51.9	1.7	21	1.3	71	1.9	50.8	0.07	53.4	0.20	91.5	0.26	509	6.9
44	Jet cooked 6 sec at 230°F with 6% NaOH	1	52.2	0.6	44	3.7	52	0.6	64.7	0.22	67.3	0.05	84.4	0.18	466	3.2
45	Jet cooked 6 sec at 230°F with 6% NaOH	2	54.9	1.6	43	3.0	50	0.7	70.0	0.10	71.4	0.19	82.9	0.19	460	4.3
28	Jet cooked 6 sec at 230°F with 6% NaOH, 5% Na_2SiO_3, & 1% H_2O_2	0	56.7	5.1	17	1.1	93	2.8	60.7	0.19	63.1	0.15	87.7	0.07	524	1.2
46	Jet cooked 6 sec at 230°F with 6% NaOH, 5% Na_2SiO_3, & 1% H_2O_2	1	51.3	1.7	45	3.9	39	2.6	69.3	0.13	71.3	0.19	80.7	0.28	420	9.3

recorded in Table IX. Since only two handsheets/set had been prepared from the all-groundwood pulps, it was necessary to use tensile test specimens of smaller size than is normally used. This can affect the test value to some extent and, accordingly, the kraft-containing controls were retested using the smaller specimen size, hence, some test values for the controls in Table IX will differ from those previously reported.

TABLE VIII

GROUNDWOOD SUSPENSION DATA FOR MEDIUM DENSITY BLEACHING SERIES

Bleach No.	Groundwood Treatment Prior to Bleaching	H_2O_2 Added, % based on fiber	pH Before Bleach	pH After Bleach	Yield, %
1	None — untreated groundwood	1	10.2	7.2	98.5
2	" " "	2	10.4	7.6	99.3
3	Jet cooked 6 sec at 230°F with 6% NaOH	1	10.8	10.2	98.3
4	Jet cooked 6 sec at 230°F with 6% NaOH	2	10.9	10.3	95.3
5	Jet cooked 6 sec at 230°F with 6% NaOH, 5% Na_2SiO_3, & 1% H_2O_2	1	10.8	10.5	95.8

In an effort to elucidate the mechanism of strength improvement resulting from jet/alkali processing, samples of untreated and jet/alkali treated pulp were examined at magnifications ranging from 40 to 430 using a standard binocular microscope. While outstanding differences were not evident, a number of rather subtle changes were observed. First of all, the jet/alkali pulp appeared to be more conformable than the untreated groundwood. The size and, possibly, number of shives was reduced in jet treatment. C-stain produced a yellow to yellow-green color in the jet/alkali pulp in contrast to an orange-yellow color in the untreated pulp which is normal for groundwood. The color developed in the jet-cooked pulp suggests that some chemical action had occurred.

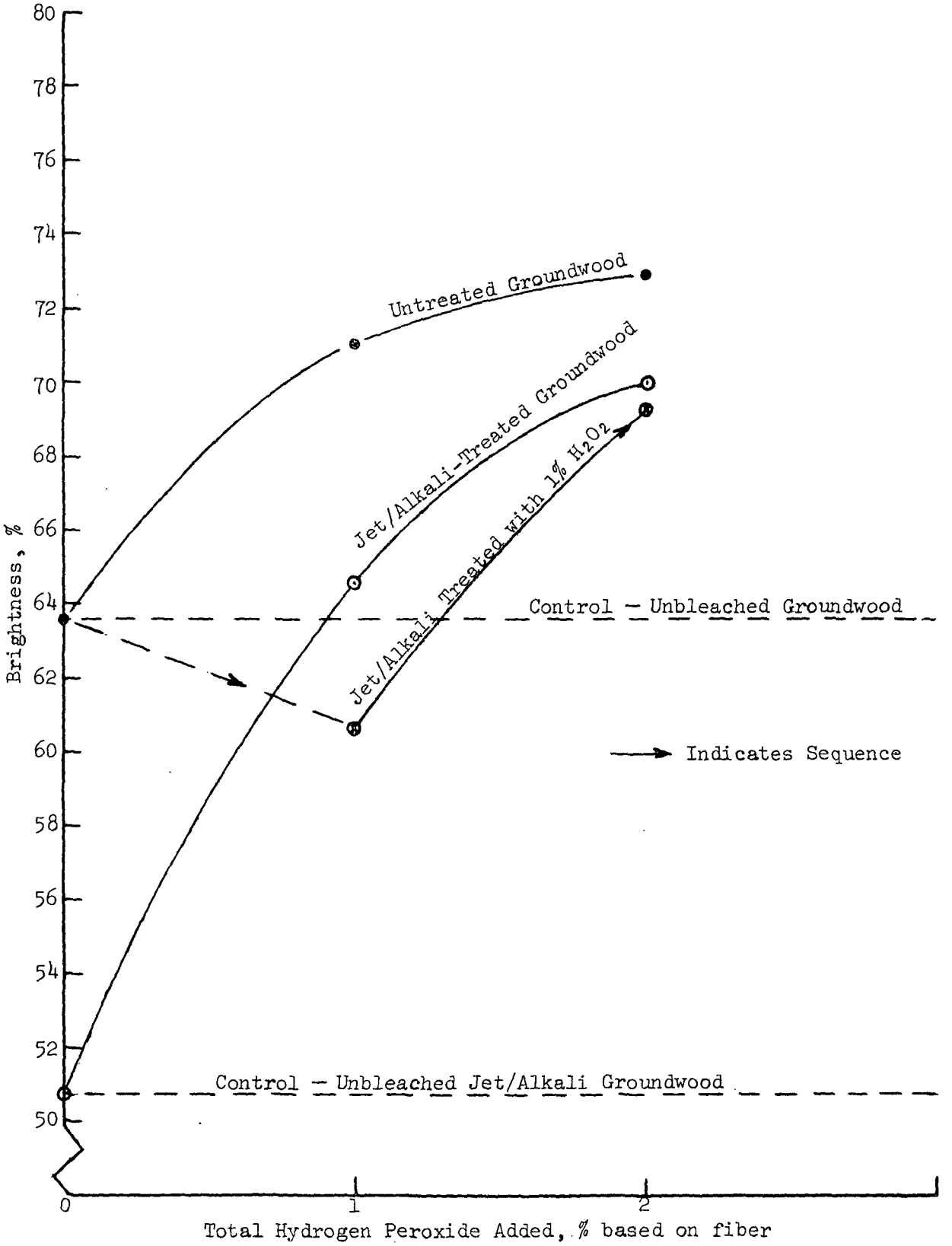

Figure 26. The Effect of Peroxide Level on the Brightness of 100% Groundwood Handsheets (Medium Density Bleaching Series)

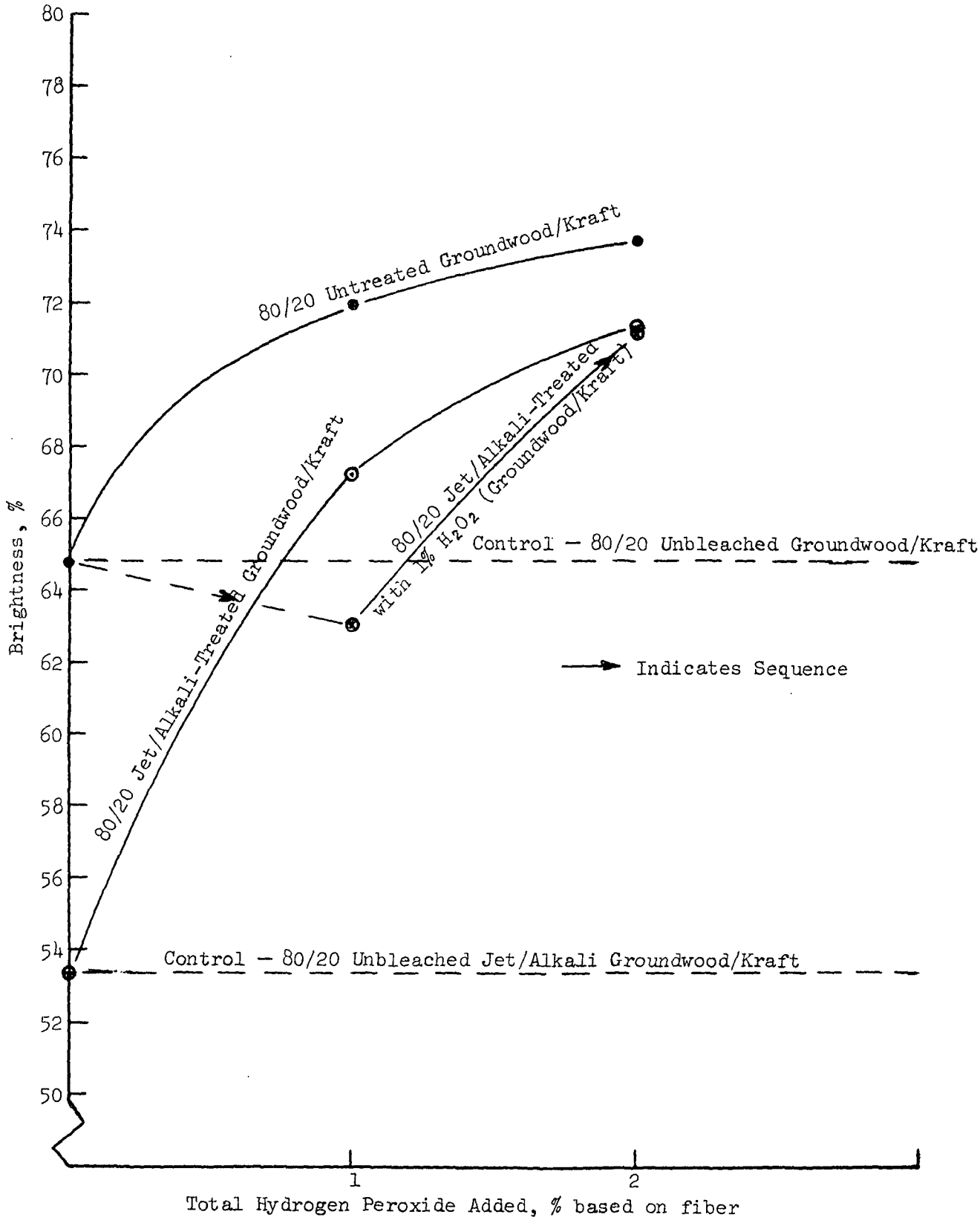

Figure 27. The Effect of Peroxide Level on the Brightness of 80/20 Groundwood/Kraft Handsheets (Medium Density Bleaching Series)

TABLE IX

THE EFFECT OF THE JET ALKALI PROCESS ON GROUNDWOOD PROPERTIES
(100% Groundwood papers)

Set No.	Description	CF of Whole Groundwood Pulp, ml	CF of 48/100 Fraction, ml	Fraction Retained on 100 Mesh, %	Basis Wt., g/M²	Thickness, µm	Density, g/cc	Breaking Length, km	T.E.A., g-cm/sq cm	Stretch, %	Tensile Stiffness, kg/cm	Tear Factor, (single sheet)	Bendtsen Air Permeability, ml/min	Brightness, %	Opacity, %	Scattering Coeff. sq cm/g
1	Control, 100% kraft	--	--	--	61.4	114	0.537	5.32	97.6	4.1	327	124.1	255	86.4	71.5	304
2	Control, 50/50 untreated groundwood/kraft	--	--	--	61.9	145	0.426	3.65	46.4	2.8	258	78.2	399	69.4	86.6	522
3-1	Control, 80/20 untreated groundwood/kraft	--	--	--	64.7	159	0.405	2.72	27.3	2.1	207	49.2	439	64.8	92.5	661
11-1	Control, 80/20 jet-treated groundwood[a]/kraft	90	--	--	63.6	119	0.533	4.80	43.5	2.0	346	51.9	71	53.4	91.5	509
3-1-A	Control, 100% untreated groundwood	150	630	22.6	62.2	160	0.389	2.24	12.5	1.3	194	19.3	558	63.6	92.9	698
11-1-A	Groundwood jet cooked 6 sec at 230°F with 6% NaOH	90	590	23.0	63.4	124	0.511	4.84	35.7	1.7	317	21.5	56	50.8	93	531
26-A	Groundwood jet cooked 6 sec at 230°F with 6% NaOH & 3% Na_2SO_3	105	610	23.8	62.3	124	0.502	5.43	42.2	1.8	366	22.8	41	54.7	91.6	482
28-A	Groundwood jet cooked 6 sec at 230°F with 6% NaOH & 1% H_2O_2	100	590	21.5	61.4	126	0.487	4.36	31.5	1.8	277	21.2	86	60.7	90.1	576
36-A	Groundwood jet cooked 6 sec at 230°F with 6% NaOH & 0.5% $NaBH_4$, pH 7[b]	90	590	23.1	63.2	125	0.506	5.04	37.0	1.7	312	22.5	50	58.2	90.3	498
37-A	Groundwood jet cooked 6 sec at 230°F with 6% NaOH & 0.5% $NaBH_4$, pH 5[c]	100	585	24.6	63.2	123	0.514	5.29	39.6	1.8	324	21.5	39	56.1	91.2	495
43-A	Control, bleached groundwood, 2% H_2O_2	150	--	--	63.9	175	0.365	2.12	16.9	1.6	181	17.8	504	73.0	90.7	716
45-A	Bleached groundwood (jet cooked 6 sec at 230°F with 6% NaOH)-2% H_2O_2	90	--	--	66.0	121	0.544	4.79	43.0	1.9	352	19.7	33	70.0	83.4	461

[a] Jet cooked 6 sec at 230°F with 6% NaOH.
[b] pH adjusted to 7 with SO_2 after jet cooking.
[c] pH adjusted to 5 with SO_2 after jet cooking.

Note: Set numbers ending in -A were prepared from 100% groundwood.
0.5% of $NaBH_4$ = 4.15% of Borol.
Strength properties listed in this table for the controls will not necessarily agree with those listed in previous tables. This is due to a change in the size of the test specimen. A smaller specimen was used in testing the all groundwood papers due to the limited supply and, hence, the controls were retested utilizing the reduced sample size.

Samples of the aforementioned pulps and the handsheets prepared therefrom were subsequently examined under the scanning electron microscope at magnifications ranging from 80 to 2000. In preparation for this work, the water-suspended groundwood fibers were air dried onto glass surfaces. The dried fibers and groundwood paper samples were then metallized with carbon and gold/palladium. Selected electron micrographs are presented in Fig. 28-33.

Figure 28. Untreated Groundwood, 300X

Figure 29. Jet/Alkali-Treated Groundwood Fibers, 300X

Figure 30. Jet/Alkali-Treated Groundwood Fibers, 1000X

Figure 31. Paper from Untreated Groundwood, 600X

Figure 32. Paper from Jet/Alkali-Treated Groundwood, 600X

Figure 33. Paper from Jet/Alkali-Treated Groundwood, 2000X

DISCUSSION OF RESULTS

Several effects are indicated in the results of the base-line study (Tables I and II). In general, the new controls compare quite favorably with those given in Report Two although the all-kraft controls tend to be of slightly lower density and strength. The set of sheets prepared from the 80/20 blend containing groundwood jet cooked 6 sec at 2% consistency with 10% NaOH (Set 6) does not quite match the strength properties of its counterpart in Report Two (Ref. 87), however, like the earlier set, it provided a breaking length which was approximately 90% of the all-kraft controls and a T.E.A. which was about 94% of the new 50/50 controls. Hence, while the new groundwood-containing papers were slightly weaker than those previously reported, their strength properties relative to the new controls remain about the same thereby confirming the effectiveness and reproducibility of the jet-alkali process.

Jet cooking at 5% consistency with 4% NaOH (Sets 5 and 7) was not as effective from the standpoint of strength properties as was the 2% consistency and 10% alkali treatment in spite of the fact that the effective alkali concentration was the same in both cases. Hence, effective alkali concentration is not indicated to be the controlling factor in strength development. Increasing the dwell time from 4 to 6 sec produced slight increases in density, breaking length, and tensile stiffness and slight decreases in stretch, air permeability, brightness, and scattering coefficient. T.E.A., tear, folding endurance, and opacity were either unaffected or not consistently affected.

As would be expected, increasing the fiber consistency to 5% at the same effective alkali concentration greatly reduced the free alkali content and lowered the pH after cooking (refer to Table II). Some decline in combined alkali was also obtained under these conditions. In general, the yield values

(93-97%) compare favorably with those previously reported but the percentage alkali accounted for is slightly lower.

In the examination of process variables (Table III; Fig. 2-23) breaking length (Fig. 3) is shown to increase as a function of alkali concentration up to the maximum level utilized (8% addition or 0.4% in solution). Tensile stiffness (Fig. 5) and folding endurance (Fig. 7) show a somewhat similar trend to breaking length at two of the three cooking temperatures. However, T.E.A. (Fig. 4) reaches a maximum at 6% alkali addition (0.3% in solution) in two of three cases and paper brightness (Fig. 10) declines below the level of the 80/20 controls at alkali additions in excess of 6%. Scattering coefficient (Fig. 12) follows a similar trend whereas opacity (Fig. 11) shows little change as a function of alkali concentration. Since brightness and scattering coefficient were reduced while opacity remained relatively constant, it is assumed that the absorption coefficient is increased in the jet/alkali process. Stretch and tear show an irregular response to alkali concentration as was previously found. With respect to cooking temperature, the 280° treatment provided marginal advantages in tear factor, brightness, and scattering coefficient (Fig. 6, 9, 10, and 12) but generally lower tensile properties than either one of both of the other processing temperatures. The 190° treatment approached or exceeded the other treatment temperatures in some properties but, in general, it did not match the 230° treatment at the higher alkali levels where maximum strength was attained. On the basis of these results, the 6% alkali level and 230°F were selected as optimum at the 6-sec dwell time recognizing that it represents a compromise in strength and optical properties. In most cases the effect of alkali concentration at the 13-sec dwell time tended to parallel that at the shorter time with respect to strength and optical properties (Fig. 13-23). In comparing dwell times, the longer time provided no

consistent advantages with the possible exception of opacity (Fig. 22). In fact, the longer dwell time proved detrimental to brightness and to strength properties at the higher alkali levels. Hence, the 6-sec jet treatment at 230°F with 6% alkali appeared to represent the optimum or near optimum condition considering all properties and the examination of brightness loss inhibitors was subsequently pursued under these conditions.

The yield values obtained in the examination of process variables (Table IV) fell in the range of 96-99% with little change indicated as a function of alkali level. This range in yield value is slightly higher than that previously reported on this project for aspen stone groundwood (1). As would be expected, free alkali increased with increase in the alkali level (Fig. 24) with little apparent difference as a function of the cooking temperature. Combined alkali appears to parallel the trend in tensile properties to the extent that both increase rapidly as the alkali level increases to about 6% and then tend to level off at higher alkali levels. The combined alkali plateau region appears to be about 40 mg/g. This is the same level found in the earlier work (1) for aspen stone groundwood processed at 2% consistency with 5, 10, and 15% alkali. Hence, combined alkali rather than alkali addition level or concentration in solution appears to be the controlling factor in strength development. This would be expected to vary with the particular groundwood pulp involved. Increasing the dwell time at 230°F had little effect on free and combined alkali which is again consistent with strength properties.

The brightness loss inhibitor series (Table V) produced several interesting effects. First of all, sodium sulfite is shown to provide improved strength properties at addition levels up to 3% (Set 26). Both groundwood and paper brightness values increased slightly beyond the 3% sulfite addition level

but tensile strength properties in the 80/20 blend tended to decline under this condition. Increasing the dwell time to 13 sec (Set 38) had little consistent effect on strength and optical properties. Three percent of sodium sulfite raised paper brightness from roughly 53 to 57% but this level was notably lower than that afforded by the 80/20 untreated groundwood/kraft controls (Set 3-1; 64.8% brightness).

Sodium borohydride produced somewhat similar effects (Sets 33-41) but the cost of this material would probably limit usage to low addition levels. The results indicate that something in excess of 0.1% of borohydride would be required to match the brightness afforded by 3% of sodium sulfite. Addition of 0.5% of borohydride improved breaking length roughly 10% and brightness was raised 6-8 units, depending upon the conditions employed. From this standpoint, 0.5% of sodium borohydride was slightly more effective than 3.0% of sodium sulfite. Strength was maximum when the borohydride was used in the form of the commercial product, Borol, and when the pH was reduced to 5 with SO_2. Brightness, on the other hand, reached maximum when the borohydride was used in the absence of the excess alkali associated with Borol. This tends to follow the results obtained in the examination of process variables where breaking length was found to increase slightly beyond the 6% alkali addition level but brightness declined linearly as a function of alkali level. The same reasoning may apply to the lower additions of sodium sulfite but to a lesser extent. In this case, the increase in strength may be related to the alkaline postsulfonation effect described by Kvisgaard (5).

In contrast to the beneficial effects on strength properties afforded by sodium sulfite and sodium borohydride, combinations of sodium silicate and hydrogen peroxide tended to reduce strength properties, possibly due to degradation of cellulosic components under the conditions employed. However, 1% of

peroxide (Set 28) increased brightness to a level approaching that of the untreated controls (Set 3-1) and 2% effectively bleached the pulp (Fig. 25). A further increase in peroxide to 3% produced only a modest increase in brightness. Comparison of these results with those given in Progress Report Two for jet processing of 2% consistency groundwood shows that less peroxide was required in the current work to reach the same brightness level due possibly to the presence of the sodium silicate and to the higher consistency and, hence, peroxide concentration. In effect, approximately one-half as much peroxide or 1.2-1.3% was required to prevent brightness loss in the current work compared to 2.5% in the work previously reported (1). Hence, sodium sulfite and sodium borohydride emerge as agents providing modest improvements in both strength and brightness, whereas hydrogen peroxide bleached the pulp at the 1.2-1.3% addition with some sacrifice in strength properties.

Yield values in the brightness loss series (Table VI) tended to be somewhat lower than was obtained in the examination of process variables but these values are approximate considering the presence of volatile and colloidal products in the process. Residual peroxide at the 1% addition level ranged from 9.3-19.7% which is somewhat less than was previously obtained at 2% fiber consistency, thereby supporting the indicated improvement in bleaching efficiency. However, poor bleaching efficiency was indicated at the higher peroxide levels based on residual levels.

Further improvements in efficiency were obtained in the medium density bleaching experiments (Tables VII and VIII, Fig. 26 and 27) where bleaching the jet/alkali pulp with 1% of peroxide increased brightness (Set 44) to a level surpassing the untreated groundwood controls (Set 3-1). Two percent of peroxide improved brightness (Set 45) to a level which was only 2-3 points lower

than that of the untreated but bleached groundwood (Set 43). Hence, in effect, jet/alkali groundwood proved more responsive than untreated groundwood to peroxide bleaching. Incorporation of 1% of peroxide in the jet cooker followed by medium density bleaching with 1% of peroxide (Set 46) did not provide any consistent advantages over straight 2% peroxide medium density bleaching although it appears that somewhat higher brightness levels may be attained in this manner at increased peroxide levels (refer to Fig. 26 and 27). Contrary to the adverse effect on strength properties of peroxide treatment in jet cooking, medium density bleaching following jet/alkali processing produced notable increases in strength properties as well as brightness. On the basis of the plotted data in Fig. 26 and 27 only 0.75-0.9% of peroxide was required to prevent brightness loss compared to 1.2-1.3% in the previous series. Hence, medium-density bleaching of the jet/alkali-treated groundwood appears to be the more acceptable approach not only from the standpoint of bleaching efficiency but also from the standpoint of strength properties. Yields of 95-98% were obtained in the medium density tests (Table VIII). This value coupled with a yield of approximately 97% in the jet/alkali process provides an overall yield of roughly 94% for the jet-cooked and bleached pulp. Considering the enhanced strength properties and, hence, the potential of substituting a greater proportion of the jet/alkali groundwood for kraft, the economics should be favorable.

Perhaps the most interesting, if not surprising, results obtained thus far are listed in Table IX covering all groundwood-content pulps and papers. Forgacs' method (4) for characterizing groundwood pulps based on the freeness of the 48/100 fraction does not seem sensitive to the jet/alkali pulps. The freeness values for this pulp fraction ranged from only 585-630 ml compared to 90-150 ml for the whole pulp. While the untreated groundwood produced the

highest freeness in each case, the differences between treated and untreated pulps were generally slight and inconsistent. The fraction retained on the 100-mesh sieve varied from only 21.5 to 24.6%, again without a consistent trend.

The breaking length and tensile stiffness properties of the jet-treated groundwood papers approached or exceeded those of the 100% kraft controls in several cases. In fact, addition of kraft pulp to the jet-processed groundwood actually reduced breaking length in several cases, although most other strength properties were enhanced by the kraft pulp. The most effective treatment with respect to strength properties was that afforded by 3% of sodium sulfite (Set 26-A). This all-groundwood paper had a breaking length which was 2-3 times that of untreated groundwood (Set 3-1-A) and a breaking length and tensile stiffness which exceeded the all-kraft paper control (Set 1). The T.E.A. of this paper equalled that of the 80/20 control containing jet-cooked groundwood and kraft (Set 11-1) and approached that of the 50/50 untreated groundwood/kraft controls (Set 2). As might be expected, those properties which depend upon fiber length such as stretch and tear tended to be lower than the kraft containing papers but nonetheless higher than those of the untreated groundwood paper. Air permeability was greatly reduced in line with the large increases in density. Perhaps the most noteworthy results of the series were provided by Set 45-A representing a bleached all-groundwood paper. This set provided a breaking length and tensile stiffness exceeding the 50/50 groundwood/kraft controls (Set 2) at essentially the same brightness, i.e., $\simeq$ 70%. Further, Set 45-A equalled the 80/20 jet-treated groundwood/kraft controls (Set 11-1) in breaking length, T.E.A., stretch, and tensile stiffness at a brightness which was approximately 17 points higher. Opacity was reduced somewhat but the resulting level was nonetheless higher than that of the all-kraft controls and only slightly lower than the 50/50 controls.

The speculation was previously made that the mechanism of strength improvement resulting from jet/alkali treatment was due to a swelling or plasticization of outer regions of the fibers and fiber elements. While this effect may occur to some extent, the electron micrographs (Fig. 29 and 30) suggest that the major effect is a greatly altered fiber structure. It appears that fibers are literally ruptured along their length and opened up to form a filmlike material which is capable of bonding together a relatively large number of nonreacted fiber elements as shown in Fig. 32 and 33. This effect is not evident in the untreated groundwood (Fig. 28 and 31). The same ruptured fiber is shown in Fig. 29 and 30 in which case layers comprising the fiber wall are apparent. Conceivably these layers may separate to form very thin films of bonding material. It would be expected that a pulp of this type would form a sheet of greatly increased density and strength but reduced porosity compared to the untreated pulp. Indeed, such has been found to be the case.

In contrast to refiner groundwood and thermomechanical treatment of wood chips where the long fiber fraction is increased and strength properties are increased accordingly, the jet/alkali process apparently develops strength through softening and rupture of fibers which then open up and span a relatively large area of nonreacted fiber elements. The area spanned by the ruptured fiber is greater than would normally be covered by the mere collapse of whole fibers into ribbons. In general, the increases in strength obtained from the jet/alkali treatment of aspen stone groundwood are greater than some reported for the thermomechanical treatment of spruce chips (2). While the thermomechanical and jet/alkali treatments are somewhat similar in their effects on tensile and brightness properties, they apparently differ in their effects on density, stretch, and tear. The jet/alkali process offers the potential of producing paper from 100% groundwood where high stretch, tear, and porosity are not required.

In review, near optimum conditions for processing 5% aspen stone groundwood in the jet/alkali process appear to fall in the range of 6-8% alkali (based on fiber), at 230°F, and at a dwell time of six seconds. Under these conditions, substantial improvements in breaking length, T.E.A., tensile stiffness, and folding endurance were attained while brightness and scattering coefficient were adversely affected. Stretch, tear factor, and opacity were relatively unaffected by the jet/alkali treatment. Actually, optimum conditions for strength improvement with aspen stone groundwood have consistently occurred at a combined alkali level of approximately 40 mg/g for all conditions of fiber consistency, temperature, alkali addition level, and effective alkali concentration examined thus far on the project. Hence, combined alkali appears to be the controlling factor in strength development.

Further improvements in strength coupled with moderate increases in brightness were attained by incorporating 3% of sodium sulfite or 0.1-0.5% of sodium borohydride into the jet cooking liquor. Notable improvements in brightness were attained at the expense of tensile strength by incorporating 1-2% of hydrogen peroxide into the bleaching liquor whereas medium density bleaching with peroxide provided greater bleaching efficiency and a moderate increase in strength. In the latter case, the bleached groundwood, in admixture with kraft, was stronger than the unbleached jet/alkali groundwood/kraft controls while brightness was increased from 53 to 71%.

Examination of 100% groundwood papers revealed that some of these papers were as strong or stronger than the 100% kraft controls with respect to breaking length and tensile stiffness while affording markedly higher opacity and scattering coefficient. Sheets prepared from the bleached groundwood provided substantially higher breaking length and tensile stiffness than the 50/50

untreated groundwood/kraft controls at the same brightness level ($\simeq$ 70%). Electron photomicrographs indicate that the jet/alkali treatment results in the rupture and unfolding of fibers which then function as bonding agents for nonreacted elements. This is in contrast to the thermomechanical treatment of wood chips where an increase in the long fiber fraction has been indicated to be the major contributor to strength improvement.

FUTURE WORK

In accordance with the provisions of the proposal on this project, Part Two of the program will tentatively be directed at jet pulping of softwood groundwood with emphasis placed on means to improve the responsiveness of the system. It is interesting to speculate as to the reasons for the difference in the responsiveness of softwood and hardwood groundwoods previously reported (1). This, of course, may be due to differences in the lignin and hemicellulose components but it may also be due to differences in physical structure or morphology. Hardwoods are known to contain fewer and smaller pits than softwoods. This would tend to restrict the entry of cooking liquor (alkali) into the lumen and, at the same time, it would tend to restrict the release of liquor. Conceivably, the sudden release of pressure at the exit of the jet results in a spontaneous expansion which cannot be accommodated by the small pits in hardwood and, as a result, the fiber ruptures as indicated in the photomicrographs. In the case of softwoods, however, the greater number and size of pits provide a safety valve effect which may accommodate the rapid expansion without rupture. Hence, higher cooking temperatures and pressures may be required for softwood to produce a similar effect. Results previously reported on this project (1) indicated that more severe processing conditions will probably be required in the jet treatment of softwood pulps and, hence, Part Two will explore the effects of higher processing temperatures, pressures, and alkali levels at 4-5% consistency. Lignin reactive chemicals and oxidants will be incorporated into the jet cooking liquor in further efforts to achieve greater responsiveness.

ACKNOWLEDGMENTS

The authors are indebted to Dr. Russell Parham, Mrs. Hilkka Kaustinen, and Miss Betty John for the microscopic analysis of groundwood pulps and paper.

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APPENDIX

ANALYTICAL METHODS

RESIDUAL NaOH

Samples of the treated pulp suspension were vacuum filtered at room temperature through Whatman No. 1 filter paper. Portions of the filtrate (100.0 ml with or 450.0 ml without NaOH pulp treatment) were titrated electrometrically to pH 2 with 0.10000N HCl (Harleco concentrate). A Beckman Model N pH meter equipped with a Corning Triple Purpose glass electrode (Cat. No. 476022) was used for this determination.

Two major inflection points were observed in plots of pH vs. volume HCl added. The first, near pH 8, was presumed to be due to free NaOH and the second, near pH 3.0 to 3.5, combined NaOH neutralized by wood acids present in the pulp or produced by the treatment. The difference in this volume of hydrochloric acid consumed at the two inflection points is used as a measure of the combined sodium hydroxide.

RESIDUAL HYDROGEN PEROXIDE

Portions of the filtrate prepared for the analysis of residual sodium hydroxide were placed in 250 ml Erlenmeyer flasks containing 100 ml of distilled water saturated with potassium iodide crystals, 3 ml of glacial acetic acid, and 5 drops of 5% ammonium molybdate solution. The liberated iodine was then titrated with 0.01N sodium thiosulfate using starch indicator near the end point. The milliequivalents thiosulfate per 100 ml filtrate was converted to milligram peroxide and divided by the total solids of the neutralized pulp suspension to obtain an estimate of unreacted peroxide in mg peroxide per 100 g solids.

Knowing the amounts of peroxide, sodium hydroxide and ovendry groundwood originally present, the residual peroxide was then expressed as a percentage of the amount added.

PULP YIELD

After the treated pulp suspension had been cooled to room temperature and neutralized to pH 5 with dilute hydrochloric acid; duplicate samples (50-60 g) were removed, weighed, and evaporated to dryness in 4 to 6 hours at 105°C in a forced draft oven. The weight of the ovendry residue was then used to calculate the total solids of the pulp slurry. A second set of duplicate samples was removed, weighed, and filtered. The filtrate was evaporated to dryness, after being combined with several portions of deionized water used to wash the fiber. The ovendry filtrate residue divided by the original slurry sample weight gives the soluble solids in the pulp suspension. These data, along with the amount of sodium hydroxide added to the raw pulp, were then used to calculate the yield of treated pulp in the absence of brightness loss inhibitors from the following equation:

$$Y\% = 100\ (1 + 1.46125f)(a-b)/a$$

where

a = total solids

b = soluble solids

f = g NaOH added per g raw pulp

The factor, $(1 + 1.46125f)$, is: 1.000, 1.0585, 1.0877, and 1.1169 at 0, 4, 6, and 8% NaOH added based on fiber weight.

The above equation was modified in cases where brightness loss inhibitors were used. For example, in the case of sodium sulfite, the equation becomes

$$Y\% = 100\ (1 + 1.46125f + 0.93g)(a-b)/a$$

where g = grams Na_2SO_3 added per gram of raw pulp. However, the yield values calculated in the brightness loss series are considered approximate due to the formation of volatile components and colloidal products which may be retained as total solids or enter the filtrate as soluble solids.